07:40:19 OCA PAD INITIATION - PROJECT HEADER INFORMATION 07/14/88

Active

Project #: E-18-654 Cost share #: Rev #: 0
Center # : R6538-0A0 Center shr #: OCA file #:
Work type : RES
Contract#: H-98048B Mod #: INIT. + Document : PO
Prime #: AMEND. #1 Contract entity: GTRC

Subprojects ? : N
Main project #:

Project unit: MAT ENGR Unit code: 02.010.112
Project director(s):
CARTER W B MAT ENGR

Sponsor/division names: NASA / MARSHALL SPACE FLT CTR, AL
Sponsor/division codes: 105 / 005

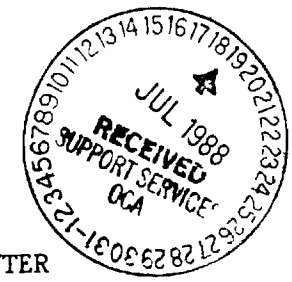
Award period: 880607 to 881206 (performance) 881206 (reports)

Sponsor amount	New this change	Total to date
Contract value	6,000.00	6,000.00
Funded	6,000.00	6,000.00
Cost sharing amount		0.00

Does subcontracting plan apply ?: N

Title: ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS- SAMPLE ANALYSIS

PROJECT ADMINISTRATION DATA



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MARSHALL SPACE FLIGHT CTR AL 35812

Security class (U,C,S,TS) : U
Defense priority rating : DO-C9
Equipment title vests with: Sponsor
NONE PROPOSED.

ONR resident rep. is ACO (Y/N): N
N/A supplemental sheet
GIT X

Administrative comments -
THIS IS A 6-MOS, FIXED-PRICE P.O. FOR THE PURPOSE OF ANALYZING GOVERNMENT-FURNISHED SAMPLES AND REPORTING ON RESULTS.

12 / 88

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

NOTICE OF PROJECT CLOSEOUT

Date 1/20/89

Project No. E-18-654 Center No. R6538-0A0

Project Director W.B. Carter School/Lab Mat. Eng.

Sponsor NASA

Contract/Grant No. P.O. H-98048B GTRC XX GIT ____

Prime Contract No. N/A

Title Electron Spectroscopy for Chemical Analysis - Sample Analysis

Effective Completion Date 12/6/88 (Performance) 12/6/88 (Reports)

Closeout Actions Required:

____ None
X Final Invoice or Copy of Last Invoice
____ Final Report of Inventions and/or Subcontracts - Already Submitted
____ Government Property Inventory & Related Certificate
____ Classified Material Certificate
____ Release and Assignment
____ Other

Includes Subproject No(s).

Subproject Under Main Project No.

Continues Project No. Continued by Project No.

Distribution:

X Project Director
X Administrative Network
X Accounting
X Procurement/GTRI Supply Services
X Research Property Management
___ Research Security Services

X Reports Coordinator (OCA)
X GTRC
X Project File
X Contract Support Division (OCA) (2)
___ Other

CONTRACTOR:	Georgia Tech Research Corporation Georgia Institute of Technology Atlanta, Georgia 30332-0420
REPORT NUMBER:	MSFC-01
TITLE OF REPORT:	Electron Spectroscopy for Chemical Analysis - Sample Analysis
DATE OF PUBLICATION:	23 August 1988
TYPE OF REPORT:	Sample Analysis
REQUISITION NUMBER:	1-7-EH-91814(1F)
AUTHOR:	W. B. Carter

PREPARED FOR:

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Marshall Space Flight Center, AL 35812

I. INTRODUCTION

This report describes the results of the ESCA analysis of the specimens listed in Table 1, which were supplied by MSFC.

TABLE 1

Specimens Analyzed with ESCA

Specimen	Exposure Conditions in Atomic Oxygen
KAPTON-4	Control (no exposure)
KAPTON-5	Exposed at 45°C, 60 W, 30 Min.
KAPTON-6	Exposed at 45°C, 60 W, 95 Min.
HDPE-19	Control (no exposure)
HDPE-20	Exposed at 45°C, 60 W, 30 Min.
HDPE-21	Exposed at 45°C, 60 W, 95 Min.
PVDF-4	Control (no exposure)
PVDF-5	Exposed at 45°C, 60 W, 30 Min.
PVDF-6	Exposed at 45°C, 60 W, 95 Min.

ESCA was performed on an SSL-100/206 Small Spot ESCA spectrometer. This instrument utilizes monochromatized Al Kα x-rays that are focused to a spot on the specimen. All data were taken with the use of a low voltage electron flood gun and a charge neutralization screen to minimize charging effects on the data. The charge neutralization screen is made of nickel and thus Ni photoionization peaks are seen in some of the spectra. Oxygen on this screen may produce small O 1s peaks. Nickel peaks and any possible contributions to the O 1s peak from the nickel screen have been neglected in the analyses that follow.

The voltage of the low energy electron flood gun was set by collecting C 1s spectra and adjusting the flood gun voltage to the nearest integral voltage setting that minimized the peak widths. Slight charging effects can be seen in a few of the spectra as a small shoulder on the low binding energy side of the effected peaks.

Because the specimens are electrically insulating and charged in the x-ray beam, it was not possible to obtain an

independent binding energy reference. The C 1s photoionization peak obtained from HDPE is often assigned a binding energy of about 285 eV. This peak can thus be used as an internal binding energy reference for the HDPE specimens. Such an approach is difficult to apply to Kapton and PVDF. The C 1s spectra of Kapton contain several component peaks that make the assignment of an internal binding energy reference difficult. Although PVDF contains CH_2 bonds (as does HDPE), the secondary substituent effects of F alter the C 1s binding energies.

The x-ray spot size and electron flood gun voltage used are recorded on the individual spectra as are the instrumental resolutions. Two resolutions were utilized, resolutions 4 and 2. Resolution 4 is the lowest resolution available on the instrument while resolution 2 is the second greatest available.

Two types of spectra were obtained for each specimen:

1. General Surveys

   General surveys were taken with the 1000 μm diameter spot (the largest available) at resolution 4 from a binding energy of -10 eV to 1090 eV. This 1100 eV range is the largest of which the spectrometer is capable.

2. High Resolution Spectra

   High resolution spectra were obtained of the most intense photoionization peaks for the major elements represented in the general surveys. These spectra were taken at resolution 2 using a 300 μm diameter x-ray spot. The high resolution spectra span a binding energy range of 20 eV.

Two types of data reduction were performed:

1. Semiquantitative Compositional Analysis

   General surveys were analyzed semiquantitatively for the atomic composition of the specimen surfaces. This was performed with the aid of the spectrometer software which takes into account relative photoionization cross sections for the atomic levels involved and the

instrument response. The effect of electron mean free path variation, which may be as large as a factor of 2, is ignored

The major photoionization peaks are labeled on the general surveys as are the major Auger peaks. The Auger peaks are indicated by an "A" following the elemental symbol.

2. Peak Fitting

The high resolution spectra were resolved into the fewest number of Gaussian components required for a reasonable fit. C 1s components were of FWHM 1.35 eV, and the O 1s components were of FWHM 1.84 eV. The widths of the N 1s and F 1s components were about 1.8 and 2.1 eV, respectively.

II. DATA ANALYSIS

A. Compositional Analysis.

The spectra collected are presented in the appendix. Table 2 lists the composition of the surface of each specimen as determined semiquantitatively from its general survey. The atomic percents are given to the nearest percent and thus may not sum to 100%.

B. Analysis of Specimens: KAPTON-4, KAPTON-5, and KAPTON-6.

The general survey from the control specimen (KAPTON-4) indicates that the specimen surface is composed primarily of C, O, and N (the constituents of KAPTON H). The C 1s spectrum of this specimen resolves into three components: 1. an aromatic component at a binding energy of about 281 eV due to the C atoms bonded to only C and H, 2. an unresolved aromatic component at about 282.2 eV, which is due to C atoms singly bonded to O or N, and 3. a component due to carbonyl type bonds (C=O). The C 1s

spectrum is similar to those for untreated KAPTON H in the published literature.[1]

There appears to be a single component to the N 1s spectrum of the unexposed specimen and two components of approximately equal magnitude to its O 1s spectrum. The O 1s component at the greatest binding energy corresponds to the ether bonded O (C-O-C) while the component at the least binding energy is due to carbonyl bonded O (C=O). O 1s spectra in the literature display a carbonyl component that is about twice the size of the ether component in contradiction to what is seen here (see footnote).

TABLE 2

Surface Composition of Polymer Specimens

Specimen	Atomic Concentration (%)			
	C	N	O	F
KAPTON-4	78	6	16	-
KAPTON-5	70	6	24	-
KAPTON-6	67	7	25	-
HDPE-19	97	-	3	-
HDPE-20	83	-	17	-
HDPE-21*	84	-	15	-
PVDF-4	56	-	1-2	42
PVDF-5	53	-	5	42
PVDF-6	54	-	5	41

* HDPE-21 had about 1% Ca on its surface.

Both general survey spectra of the plasma exposed specimens (KAPTON-5 and KAPTON-6) indicate an increased O concentration relative to the unexposed specimen. The C 1s spectra of these specimens consist of several components: 1. two components at low binding energies, which are due to the C-C-C, and the C-O and C-N bonds in KAPTON H, and 2. possibly three components at greater

[1] M. Kogoma and G. Turban, Plasma Chemistry and Plasma Processing, 6 (4), (1986) 349.

binding energies, which are due to C atoms in increased oxidation states. The proportion of C atoms in the greater oxidation states, i.e., greater than those of C-C-C, C-O, and C-N, is larger after plasma exposure that before. These three components represent about 89 percent of the C 1s signal prior to plasma exposure and about 64 percent after plasma exposure.

The N 1s spectra taken from the plasma exposed KAPTON H specimens appear to be comprised of two components. The separation between the resolved peaks is about 1.1 eV. The component at the greater binding energy may be due N-O bonds.

The O 1s spectra from the plasma exposed specimens resolve into two components of approximately equal magnitude. The existence of two resolvable components after exposure to an RF oxygen plasma is consistent with the literature as is the fact that their relative magnitudes are unchanged by the plasma (see footnote).

C. Analysis of Specimens: HDPE-19, HDPE-20, and HDPE-21.

The general survey from the control specimen (HDPE-19) indicates that the specimen surface is primarily composed of C and O. As there should be no O in the pure polymer, the O may be due to a slight oxidation of the surface and/or contamination. The solvent used to clean the specimen may have been the source of the O observed. The C 1s spectrum of this specimen contains two (2) components: 1. a major peak due to CH_2 bonds in HDPE that appears at a binding energy of about 280 eV (shifted due to charging) and 2. a small peak shifted upwards in binding energy from the major peak by about 4.2 eV that is probably due to oxidation of C.

Both general survey spectra of the plasma exposed specimens (HDPE-20 and HDPE-21) indicate an increased O concentration relative to the unexposed specimen. Both the relative and absolute increase in surface O concentration after plasma exposure are greater for the HDPE than for either KAPTON H or PVDF (see TABLE 2).

The C 1s spectra of the plasma exposed specimens consist of several components: 1. a major component due to $\underline{C}H_2$ bonds, and 2. several components at larger binding energies that correspond to C in various states of oxidation. The differences between the spectra from these two specimens cannot be considered significant since only one spectrum of each type was taken from each specimen.

D. Analysis of Specimens: PVDF-4, PVDF-5, and PVDF-6.

The general survey from the control specimen (PVDF-4) indicates that the specimen surface is composed of primarily F and C. There is a minor amount of O, which is probably due to contamination.

The C 1s spectrum of the unexposed specimen consists of two resolvable components separated by about 4.5 eV. That at the lowest binding energy is due to $\underline{C}H_2$ and that at the highest binding energy is due to $\underline{C}F_2$. It appears from the FWHM of these components that each is probably composed of several unresolved components due to various nonideal structures in the polymer, eg., $\underline{C}HF$.

The F 1s spectrum of the unexposed specimen is fit well with a single Gaussian peak. The O 1s spectrum resolves into two components separated by 1.0 eV.

Both general survey spectra of the plasma exposed specimens (PVDF-5 and PVDF-6) indicate an increased O concentration relative to the control specimen.

The C 1s and F 1s spectra of the plasma exposed specimens are similar to those of the unexposed specimen. The separation between these photoionization peaks also is unchanged by plasma exposure. The O 1s spectra of the plasma exposed specimens resolve into two components of similar magnitude and separated by about 1.4 eV.

From this data, it is not possible to determine whether the oxidation state of C is changed by plasma exposure.

III. SUMMARY

Plasma treatment appears to oxidize all of the polymers examined. Evidence for this are the increases in O on the specimen surfaces after plasma exposure and the increases in the relative amounts of C in various oxidized states following exposure.

Although the relative oxygen increase of the PVDF was greater than that of the KAPTON H specimen, the absolute increase for the PVDF was the smallest observed (see TABLE 2 and recall that KAPTON H contains structural O). The PVDF specimens displayed the least O both prior to plasma exposure and after plasma exposure. Absolute amounts of O due to plasma exposure increase in the order: PVDF < KAPTON < HDPE.

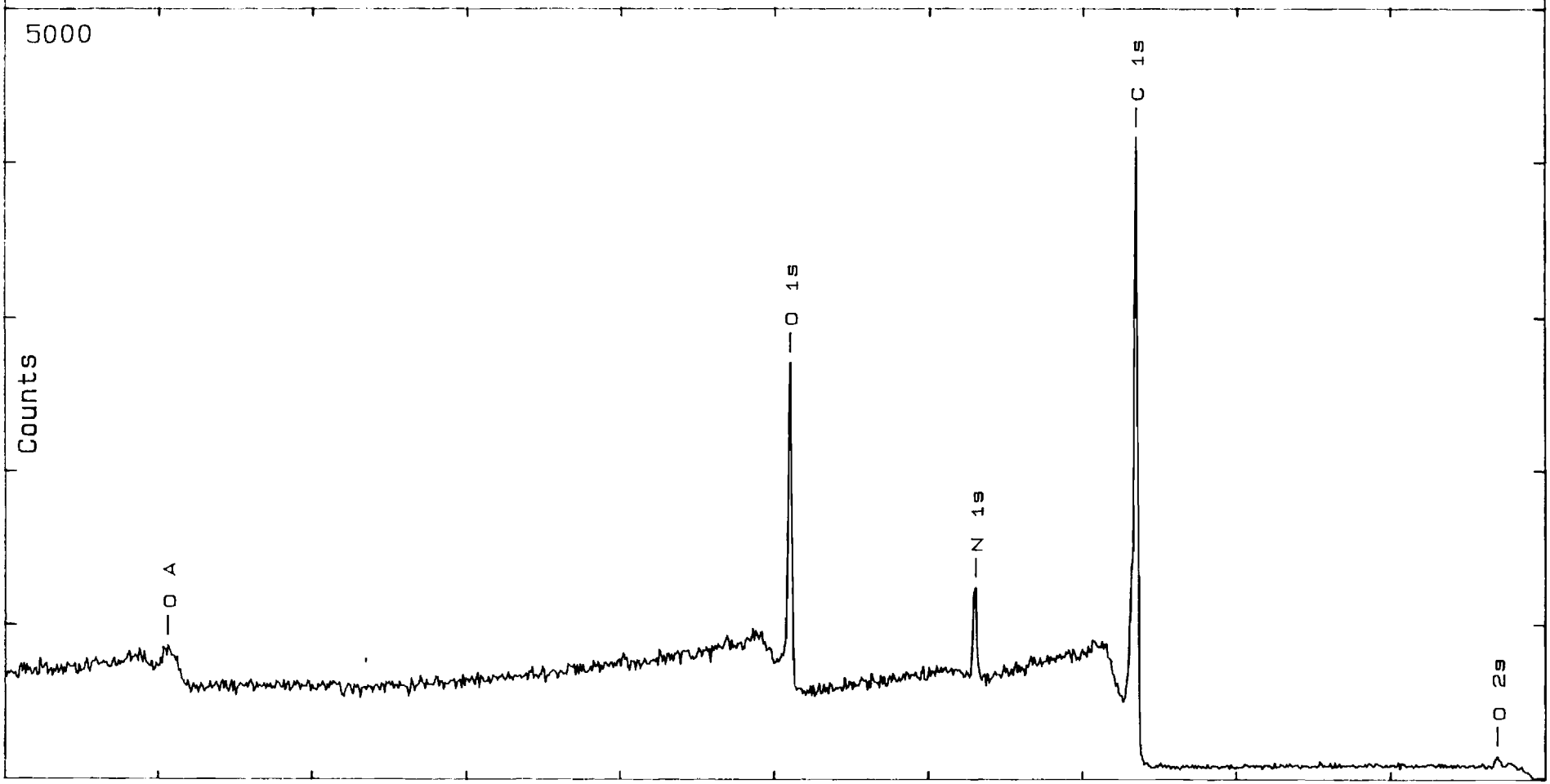
APPENDIX

ESCA SPECTRA

File: KPTNMRS12	Date: 8/16/1988	Spot Size: 1000 u	Flood Gun: 1.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4

Description: KAPTON-4, CONTROL (NO EXPOSURE)
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

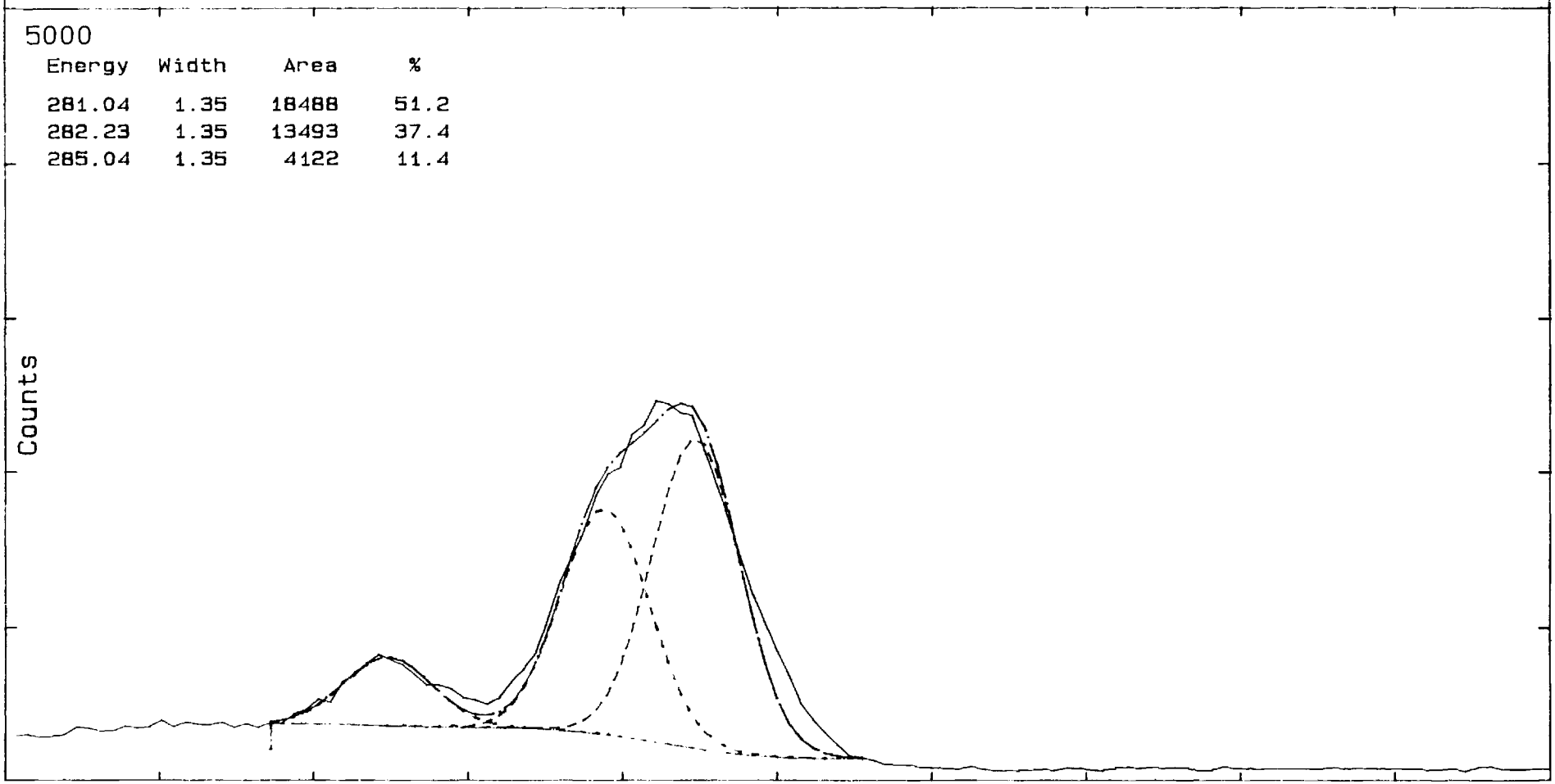


Report #: MSFC-01

File: KPTNMRS12	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 1.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: KAPTON-4 (CONTROL), C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

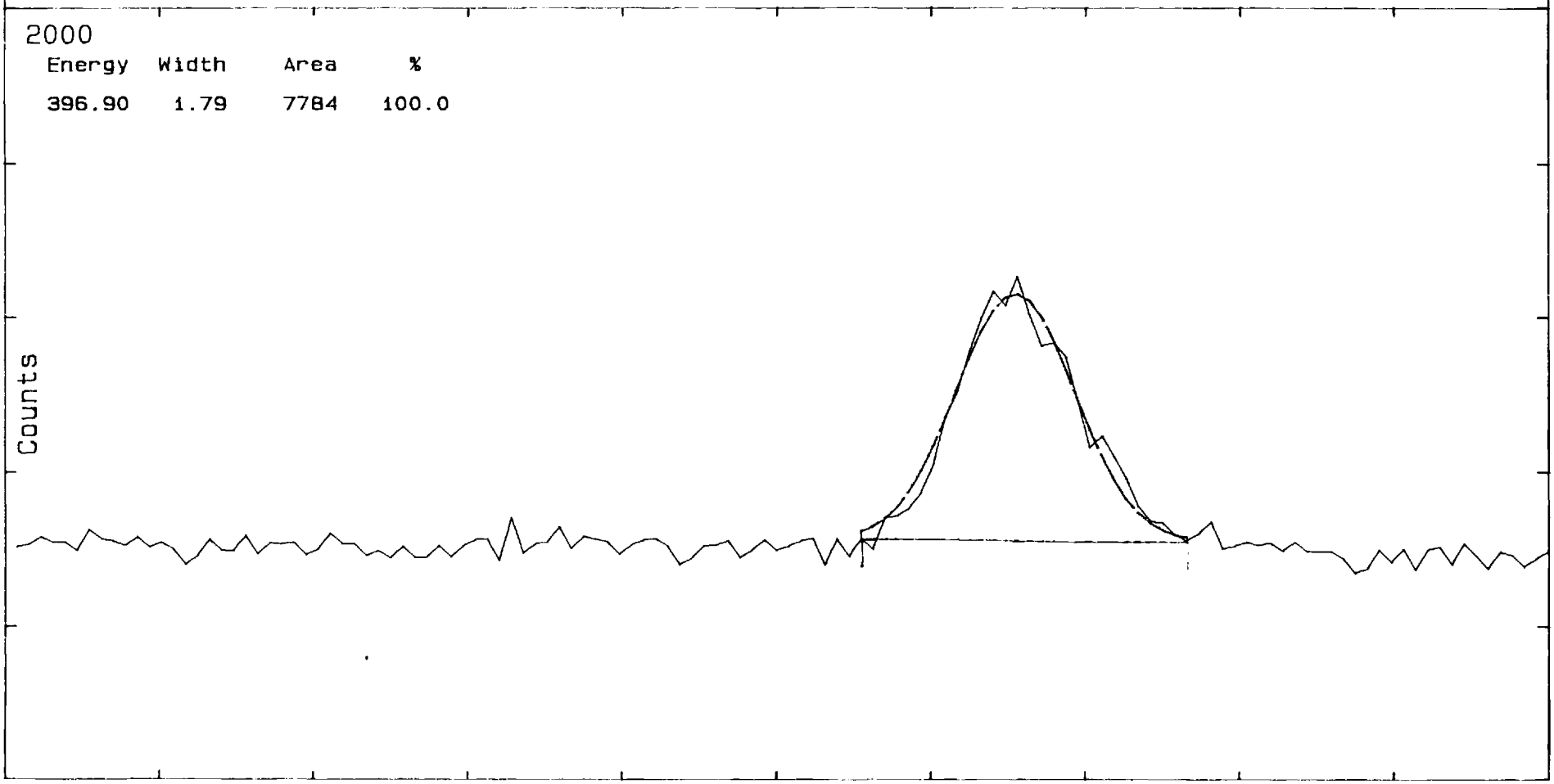


Report #: MSFC-01

File: KPTNMRS12	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 1.0 eV
Region 3	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: KAPTON-4 (CONTROL), N 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

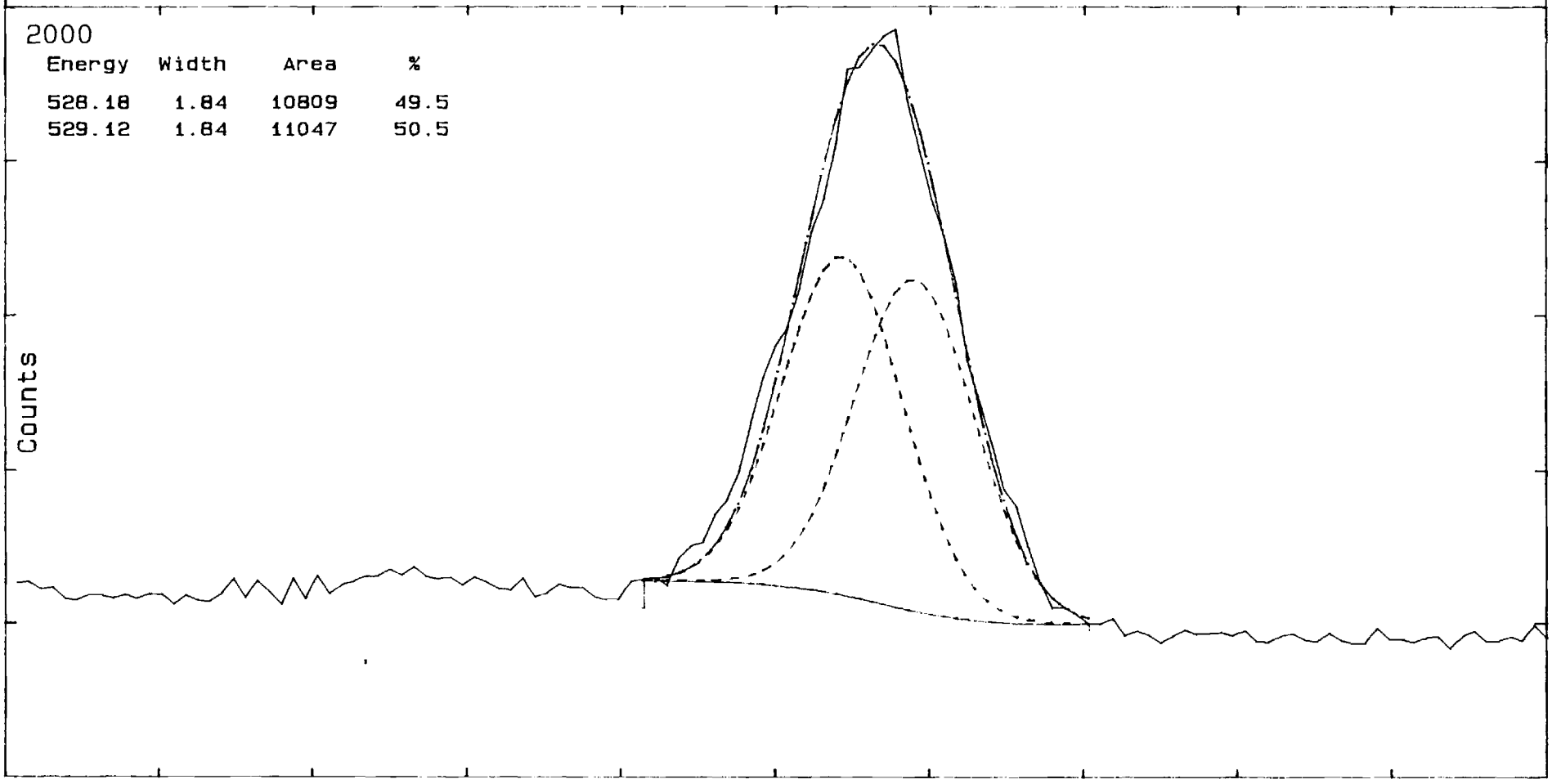


Report #: MSFC-01

File: KPTNMRS12	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 1.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2

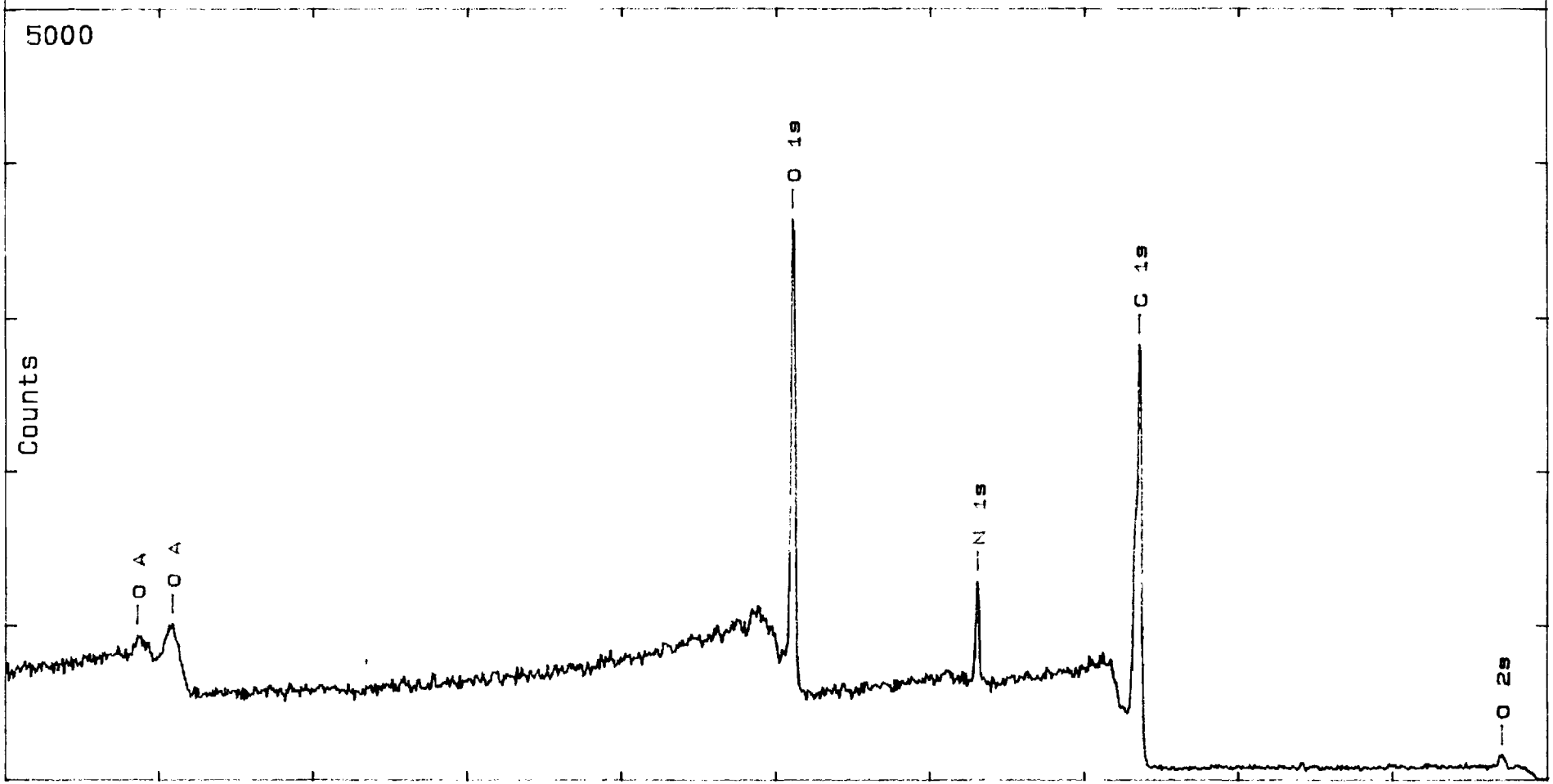
Description: KAPTON-4 (CONTROL), O 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

File: KPTNMRS13	Date: 8/16/1988	Spot Size: 1000 u	Flood Gun: 2.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4
Description: KAPTON-5, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN. CHARGE NEUTRALIZATION SCREEN			Operator: WBC

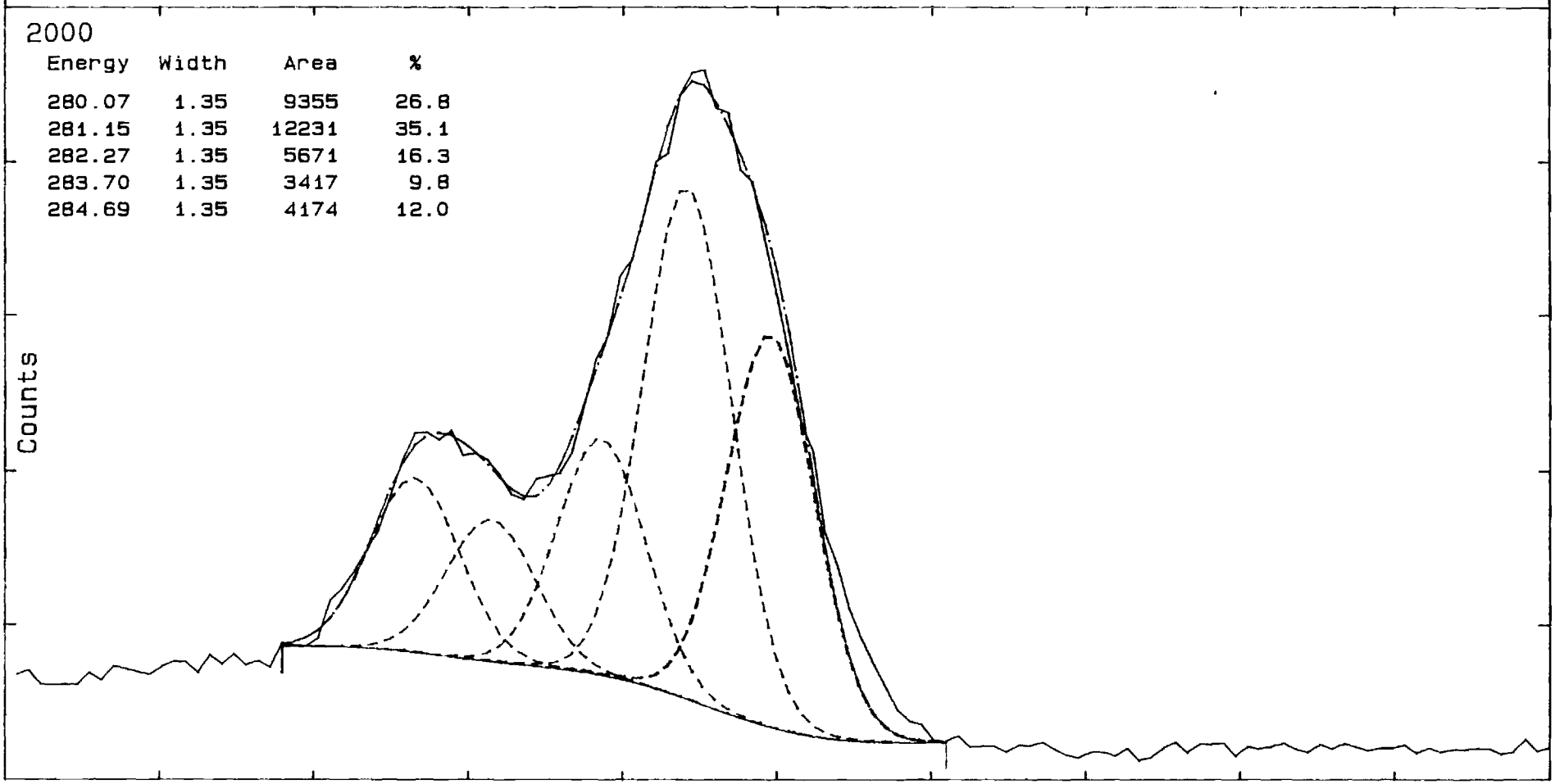


Report #: MSFC-01

File: KPTNMRS13	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: KAPTON-5, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

File: KPTNMRS13	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 3	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: KAPTON-5, N 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

2000

Energy	Width	Area	%
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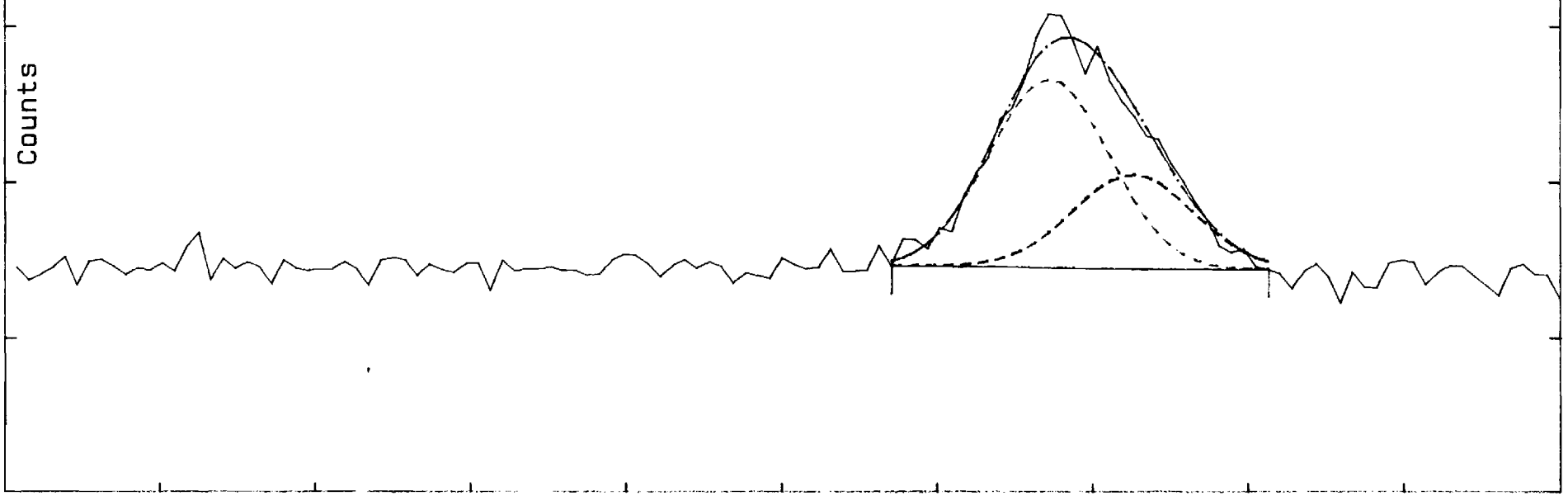
396.56	1.80	5926	66.8



410.0 Binding Energy (eV) 390.0

Report #: MSFC-01

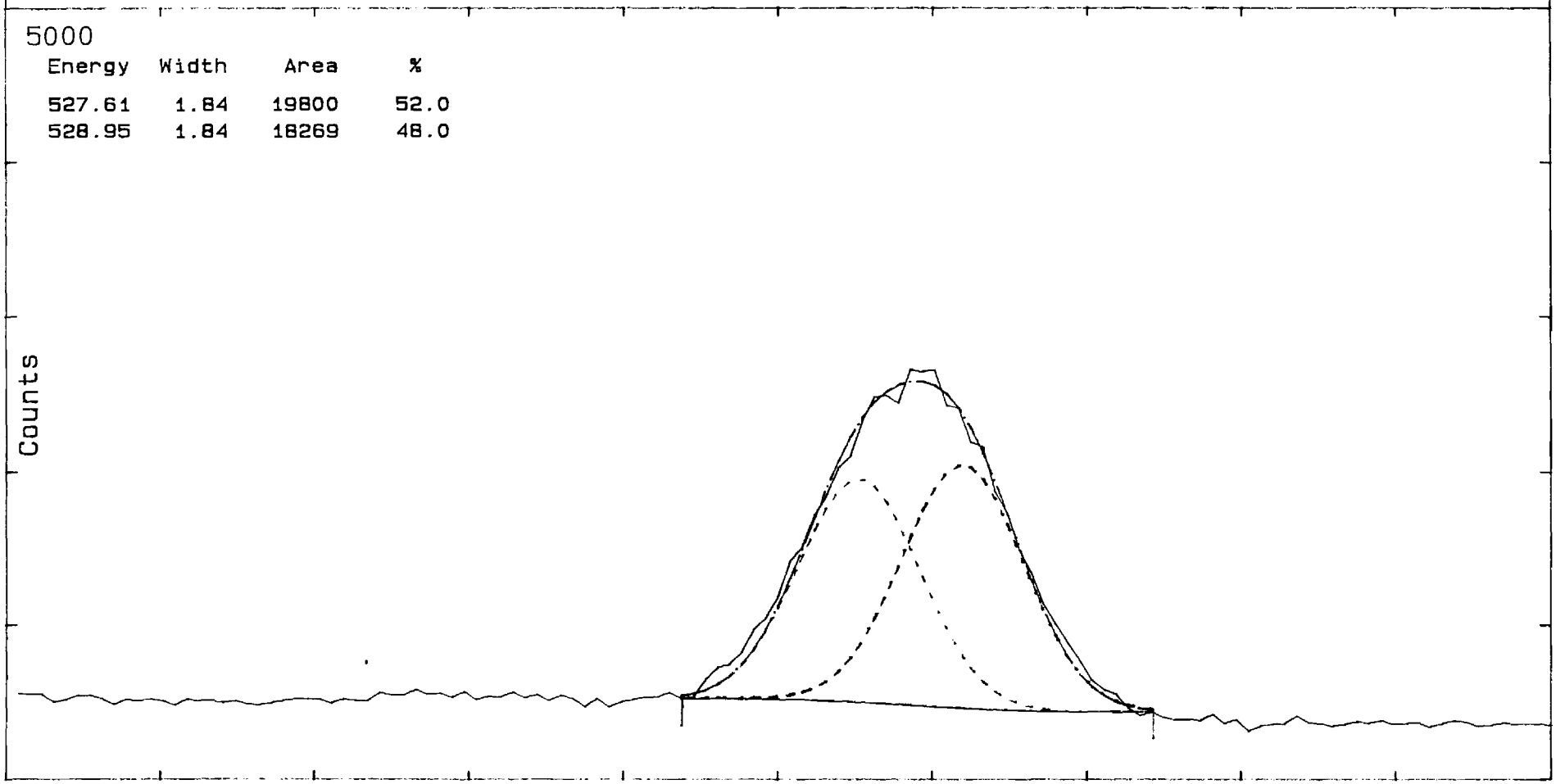
File: KPTNMRS13	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: KAPTON-5, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

5000

Energy	Width	Area	%
527.61	1.84	19800	52.0
528.95	1.84	18269	48.0



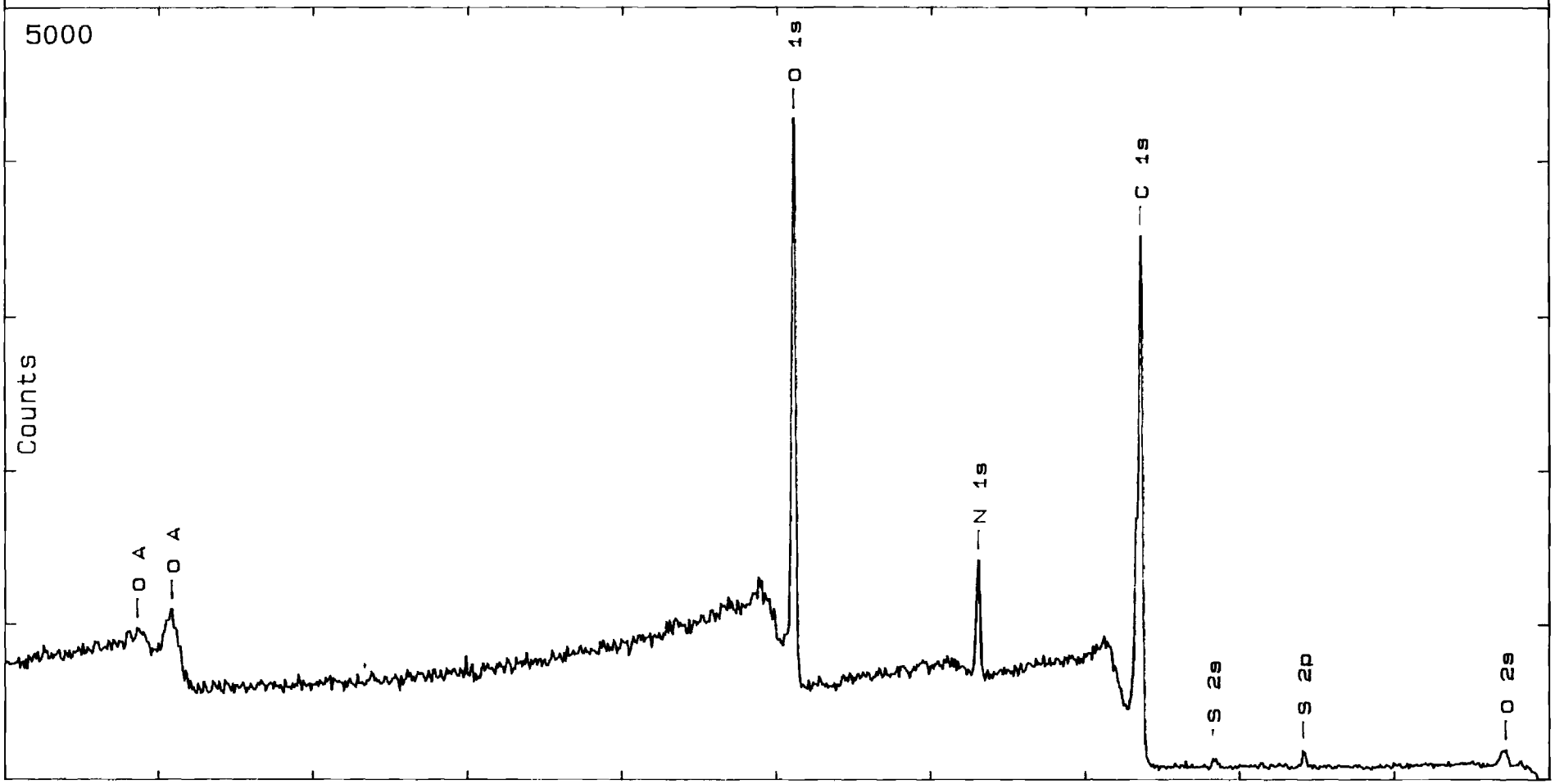
540.0 Binding Energy (eV) 520.0

Report #: MSFC-01

File: KPTNMRS14	Date: 8/17/1988	Spot Size: 1000 u	Flood Gun: 2.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4

Description: KAPTON-6, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN. CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

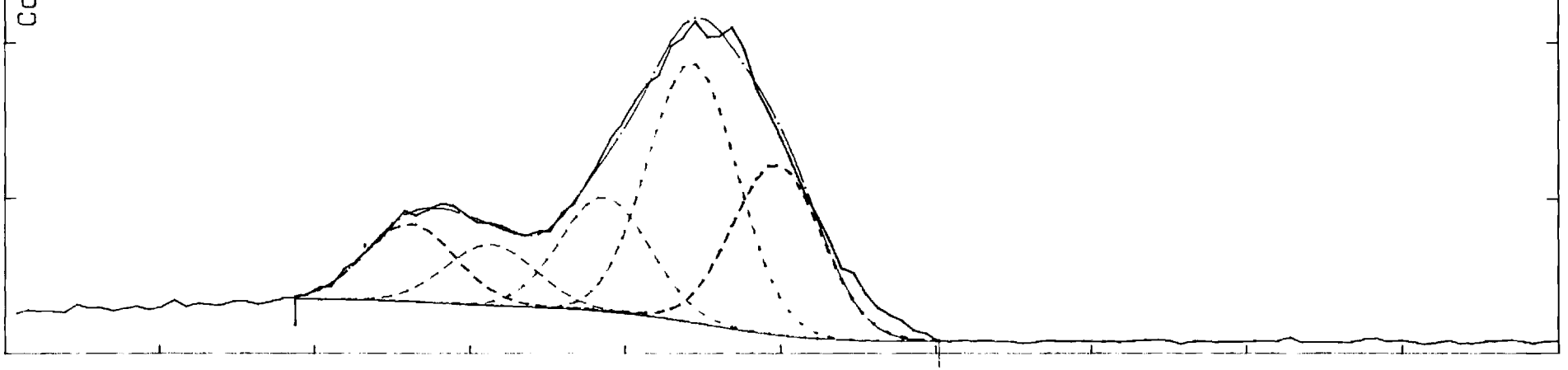
File: KPTNMRS14	Date: 8/17/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: KAPTON-6, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

5000

Energy	Width	Area	%
280.05	1.35	10103	25.0
281.13	1.35	15375	38.0
282.26	1.35	6774	16.8
283.74	1.35	3616	8.9
284.77	1.35	4567	11.3



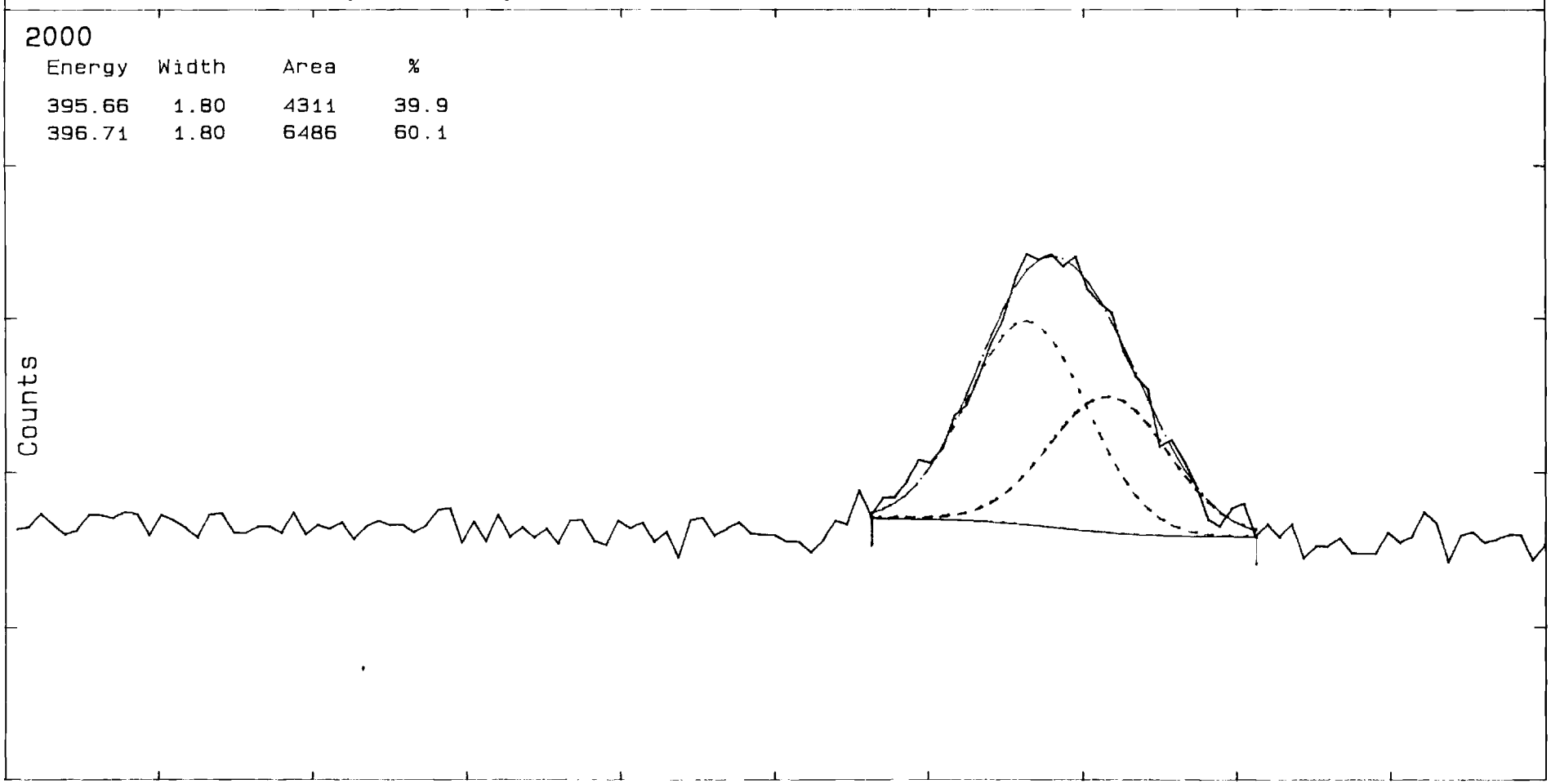
290.0 Binding Energy (eV) 270.0

Report #: MSFC-01

File: KPTNMRS14	Date: 8/17/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 3	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: KAPTON-6, N 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

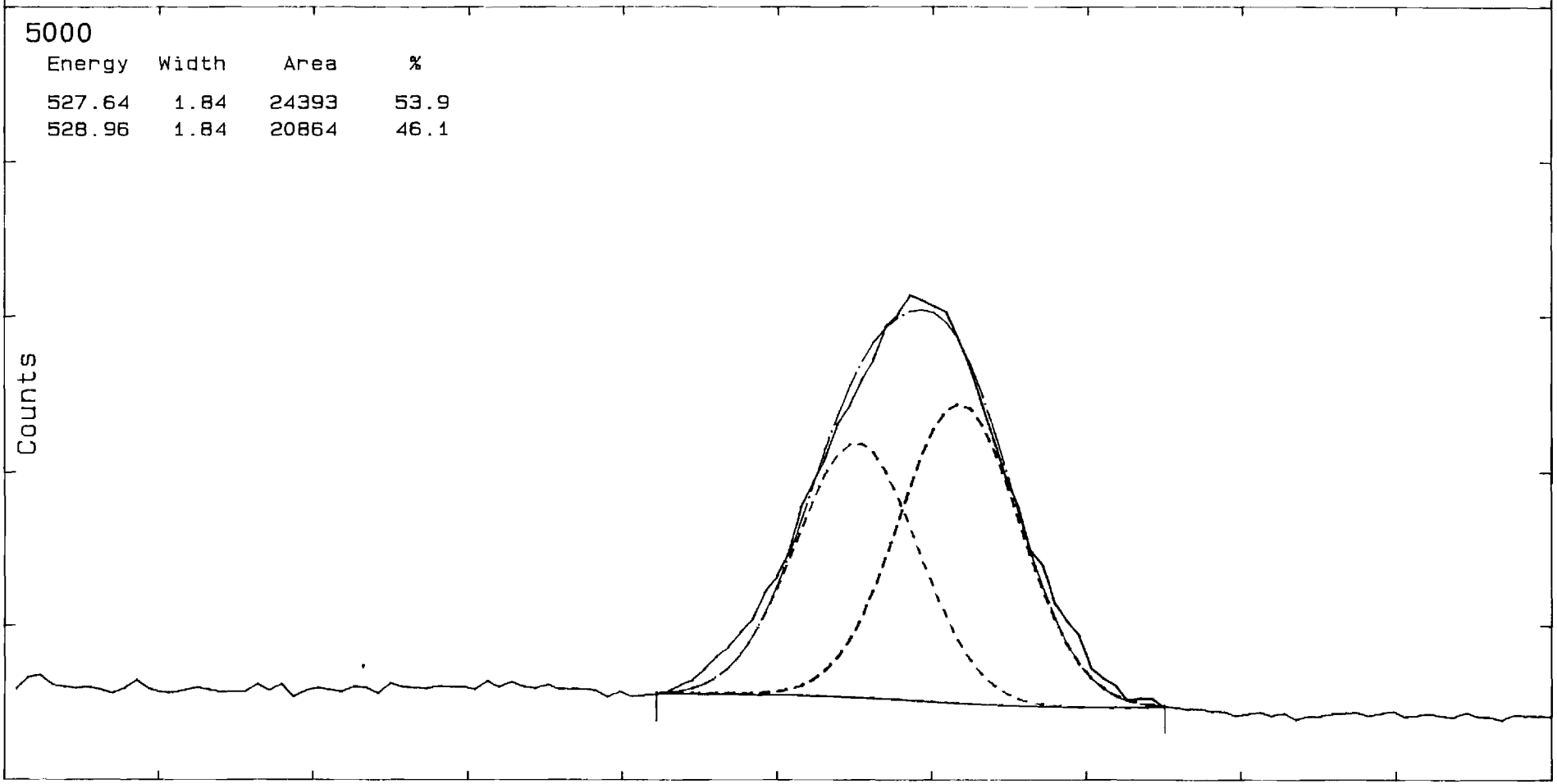


Report #: MSFC-01

File: KPTNMRS14	Date: 8/17/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: KAPTON-6, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

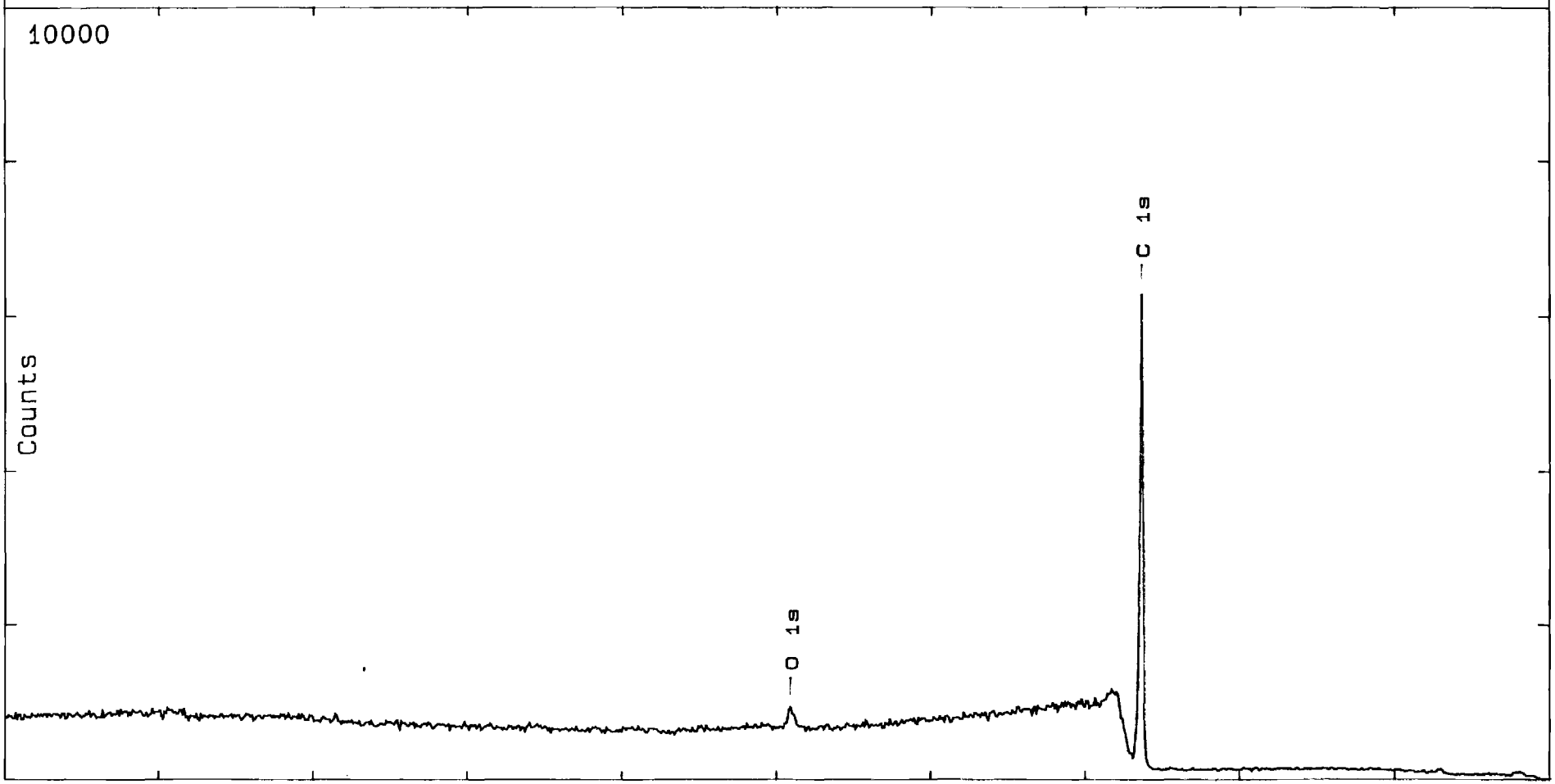


Report #: MSFC-01

File: HDPE10	Date: 8/15/1988	Spot Size: 1000 u	Flood Gun: 2.0 eV
	Disc: NASA01	# of Scans: 1	Resolution: 4

Description: HDPE-19, CONTROL (NO EXPOSURE)
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

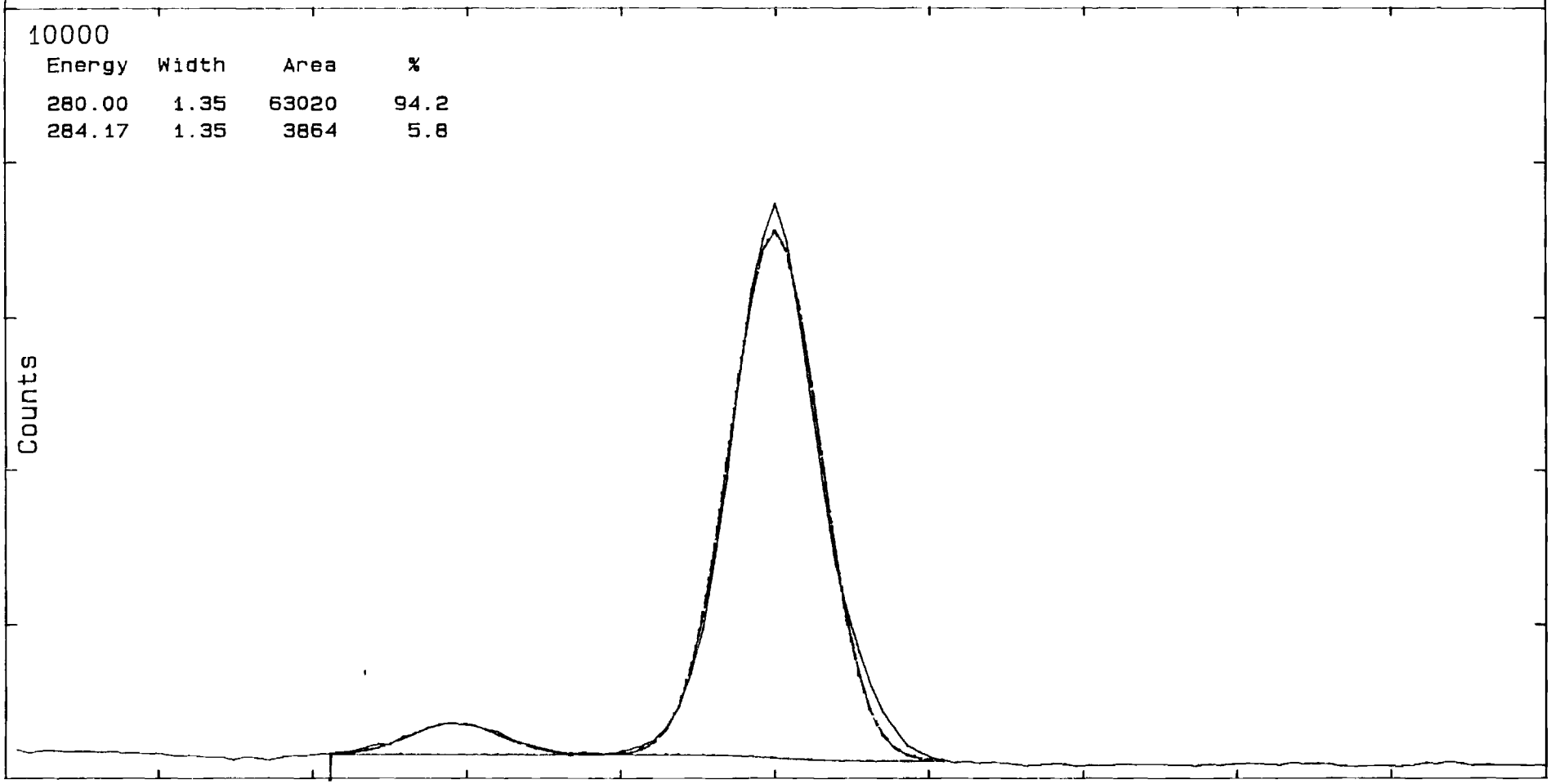


Report #: MSFC-01

File: HDPEMRS11	Date: 8/15/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 1	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: HDPE-19 (CONTROL), C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

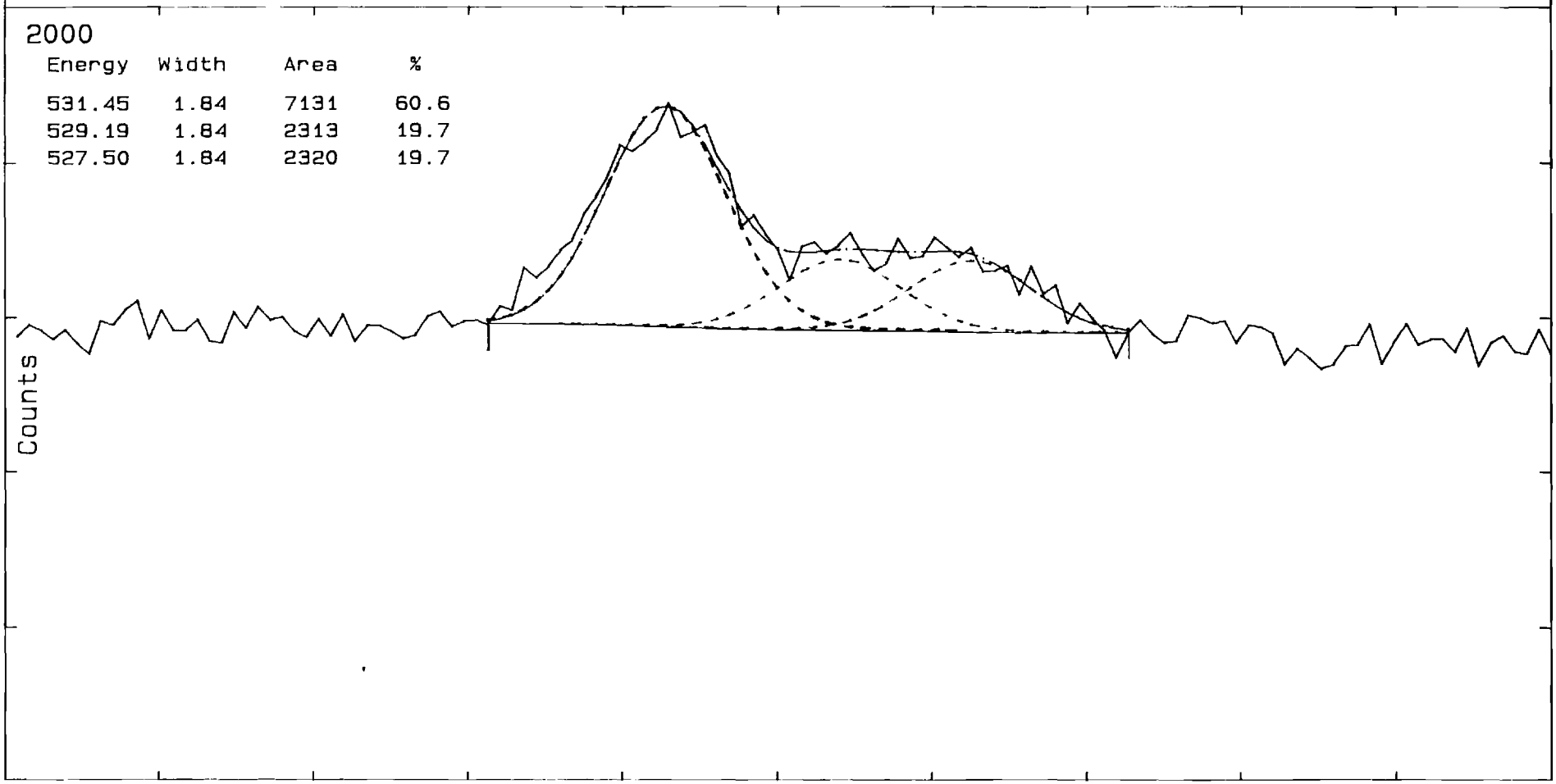


Report #: MSFC-01

File: HDPEMRS11	Date: 8/15/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 2	Disc: NASA01	# of Scans: 30	Resolution: 2

Description: HDPE-19 (CONTROL), O 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

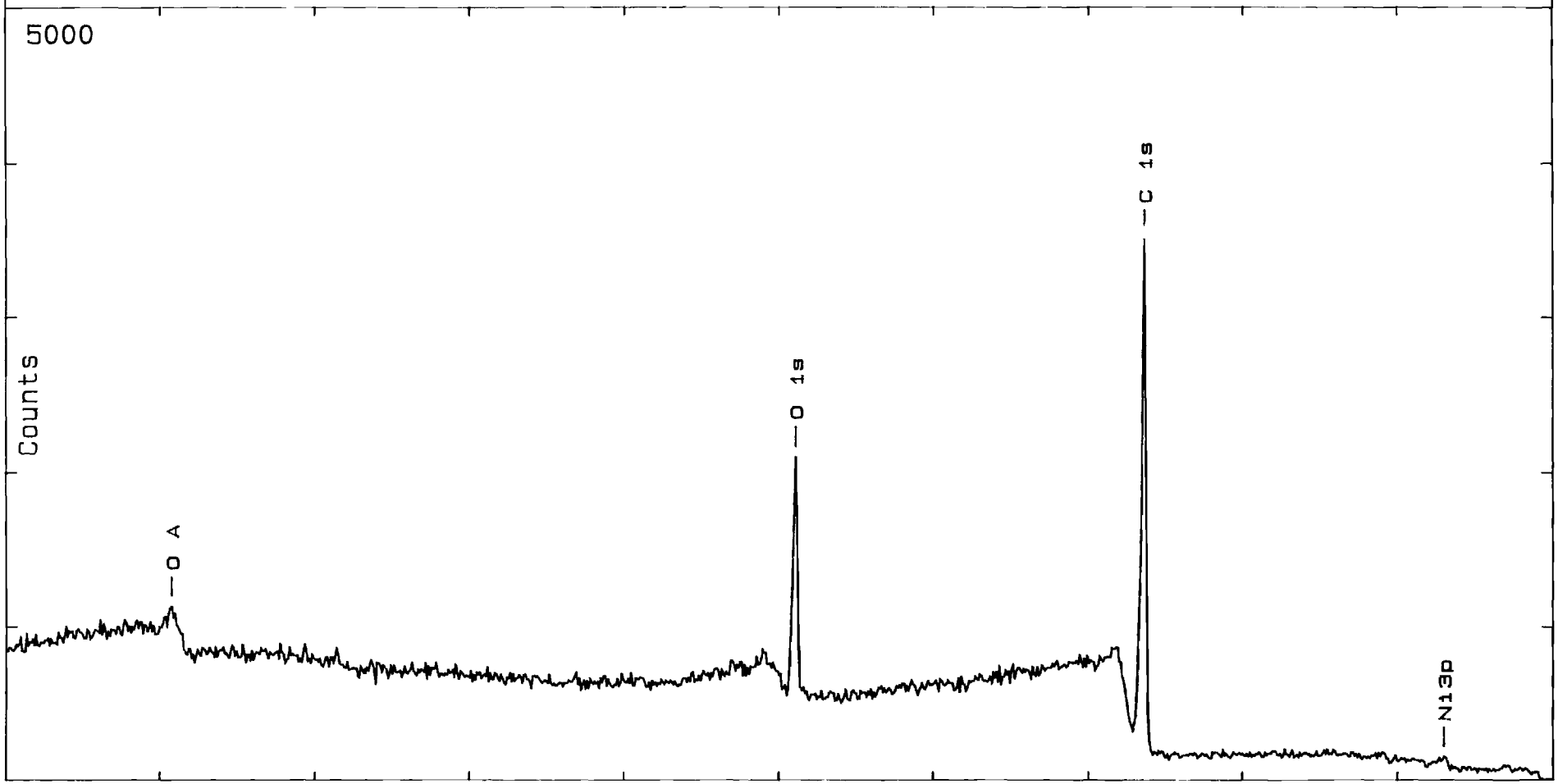


Report #: MSFC-01

File: HDPE12	Date: 8/15/1988	Spot Size: 1000 u	Flood Gun: 2.0 eV
	Disc: NASA01	# of Scans: 1	Resolution: 4

Description: HDPE-20, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN.
CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

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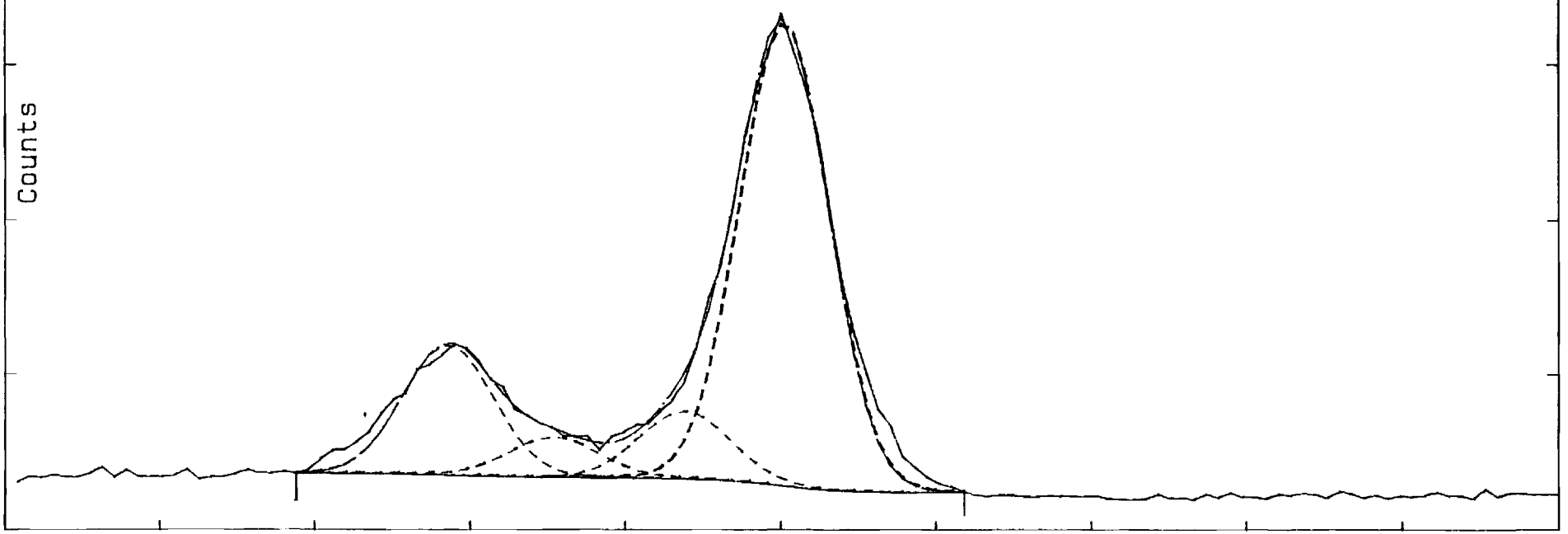
Region 1	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: HDPE-20, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN.; CHARGE NEUTRALIZATION SCREEN

Operator: WBC

5000

Energy	Width	Area	%
279.96	1.35	27340	66.2
281.25	1.35	3970	9.6
282.94	1.35	2329	5.6
284.29	1.35	7683	18.6



290.0 Binding Energy (eV) 270.0

Report #: MSFC-01

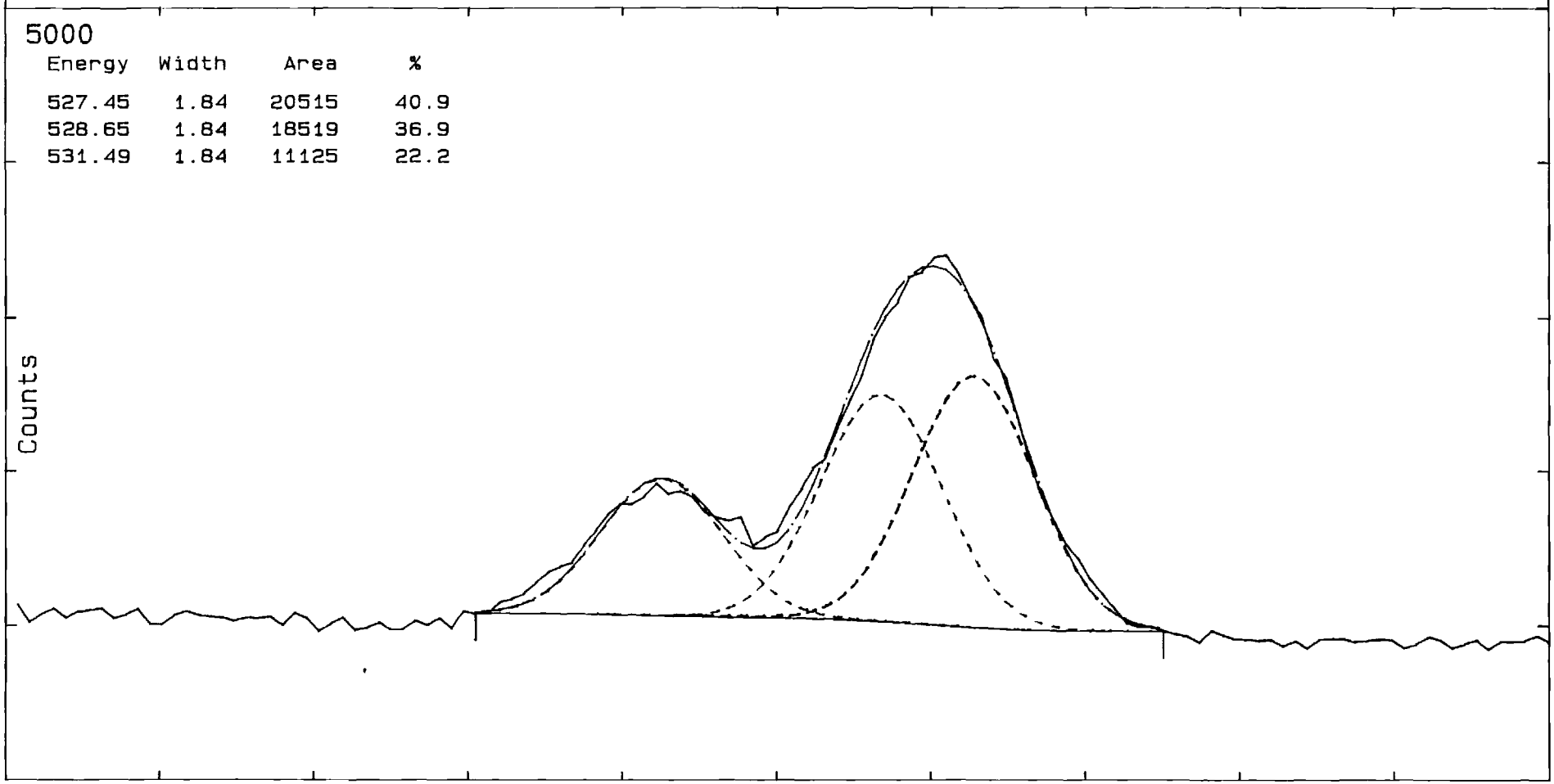
File: HDPEMRS13	Date: 8/15/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 2	Disc: NASA01	# of Scans: 30	Resolution: 2

Description: HDPE-20, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN.; CHARGE NEUTRALIZATION SCREEN

Operator: WBC

5000

Energy	Width	Area	%
527.45	1.84	20515	40.9
528.65	1.84	18519	36.9
531.49	1.84	11125	22.2

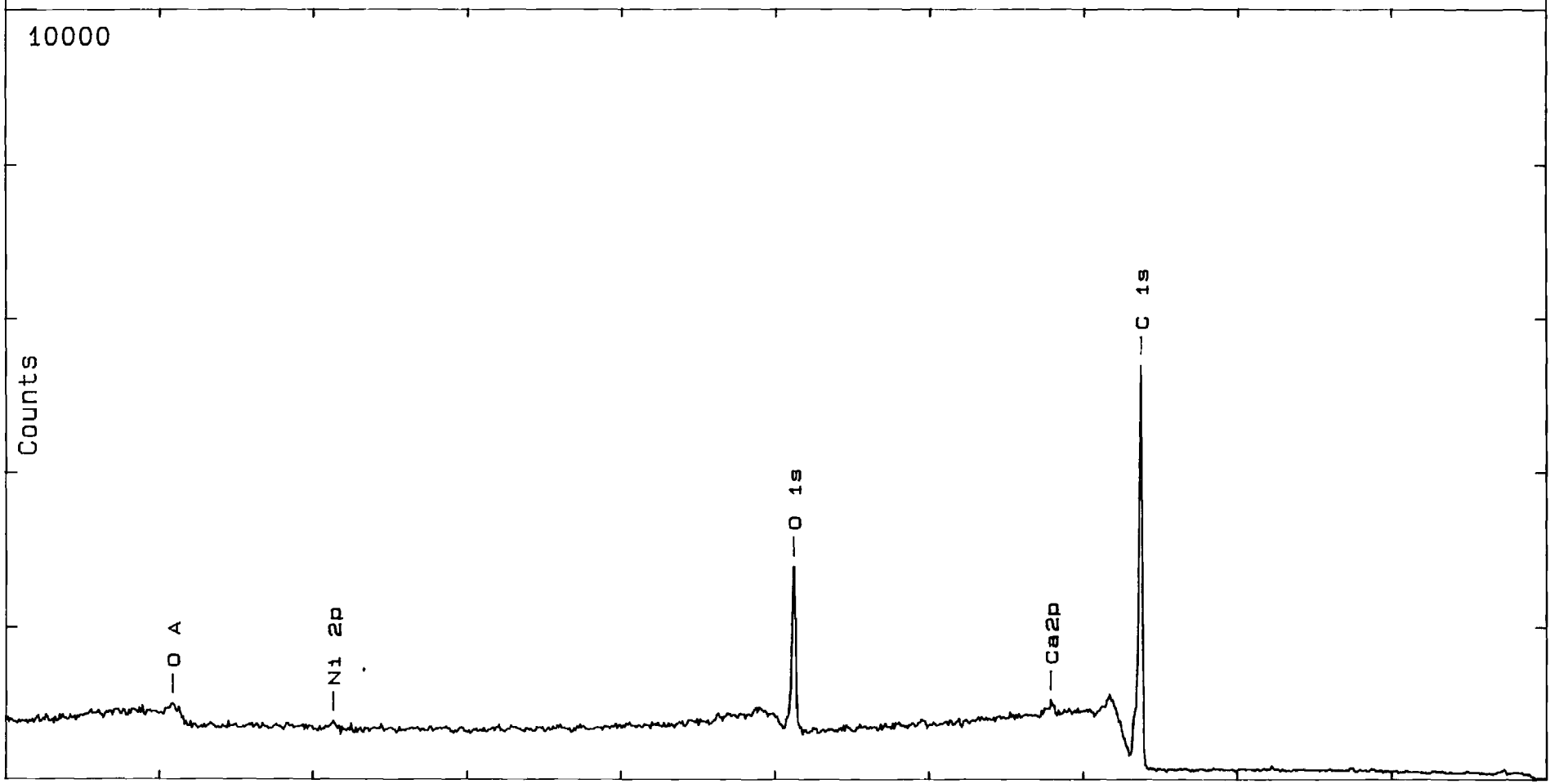


540.0 Binding Energy (eV) 520.0

Report #: MSFC-01

File: HDPE14	Date: 8/15/1988	Spot Size: 1000 u	Flood Gun: 3.0 eV
	Disc: NASA01	# of Scans: 1	Resolution: 4

Description: HDPE-21, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN. Operator: WBC
CHARGE NEUTRALIZATION SCREEN



Report #: MSFC-01

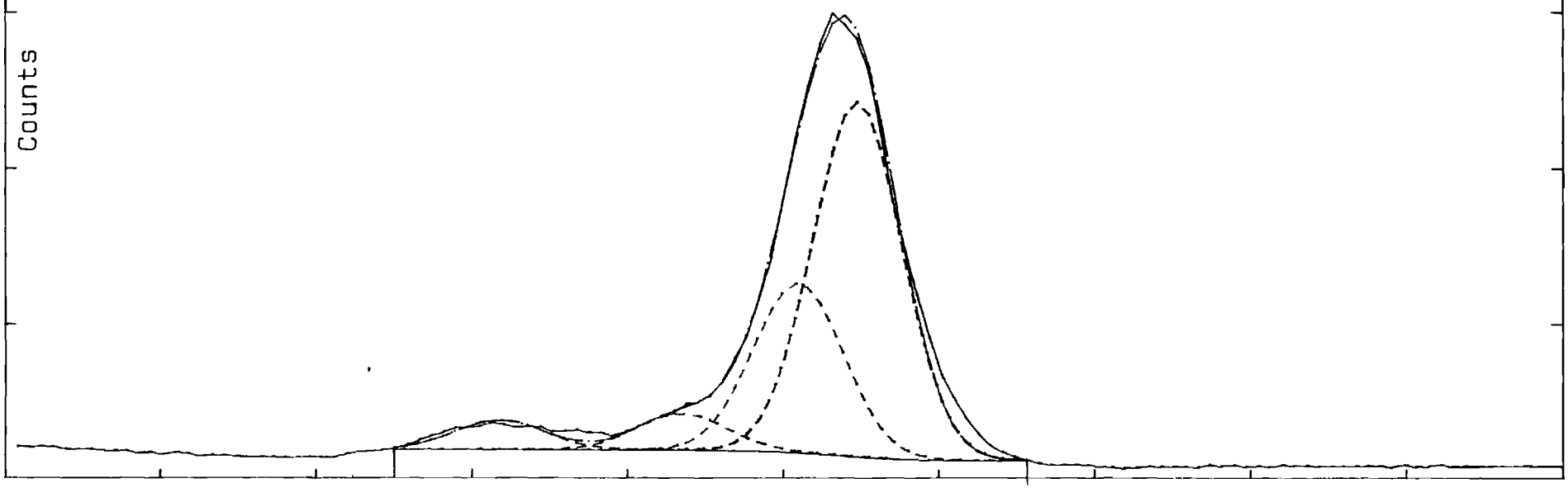
File: HDPEMRS15	Date: 8/15/1988	Spot Size: 300 u	Flood Gun: 3.0 eV
Region 1	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: HDPE-21, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN.; CHARGE NEUTRALIZATION SCREEN

Operator: WBC

10000

Energy	Width	Area	%
279.05	1.35	42041	59.9
279.81	1.35	20234	28.9
281.31	1.35	4345	6.2
283.65	1.35	3511	5.0



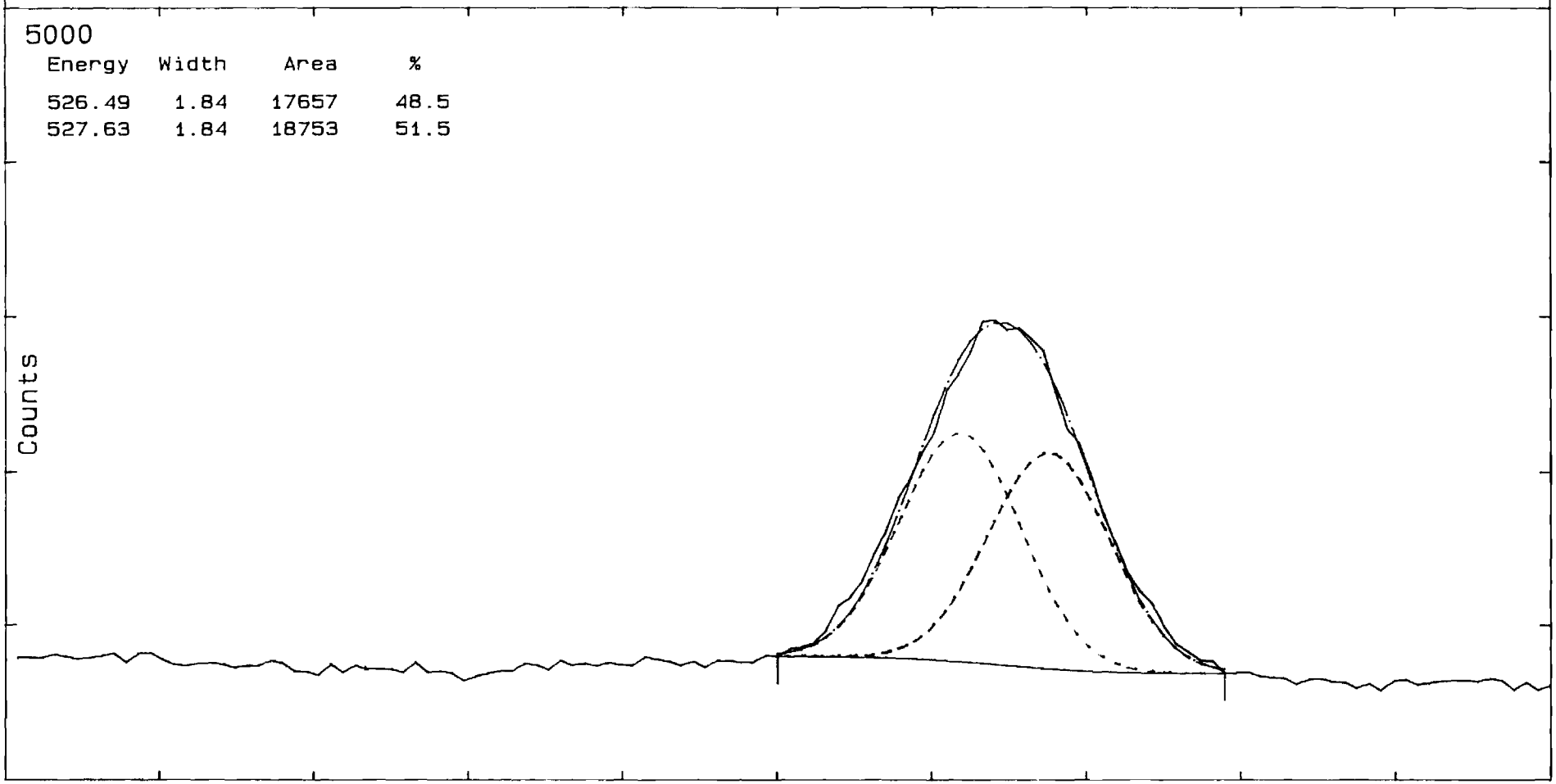
290.0 Binding Energy (eV) 270.0

Report #: MSFC-01

File: HDPEMRS15	Date: 8/15/1988	Spot Size: 300 u	Flood Gun: 3.0 eV
Region 2	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: HDPE-21, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN.; CHARGE NEUTRALIZATION SCREEN

Operator: WBC

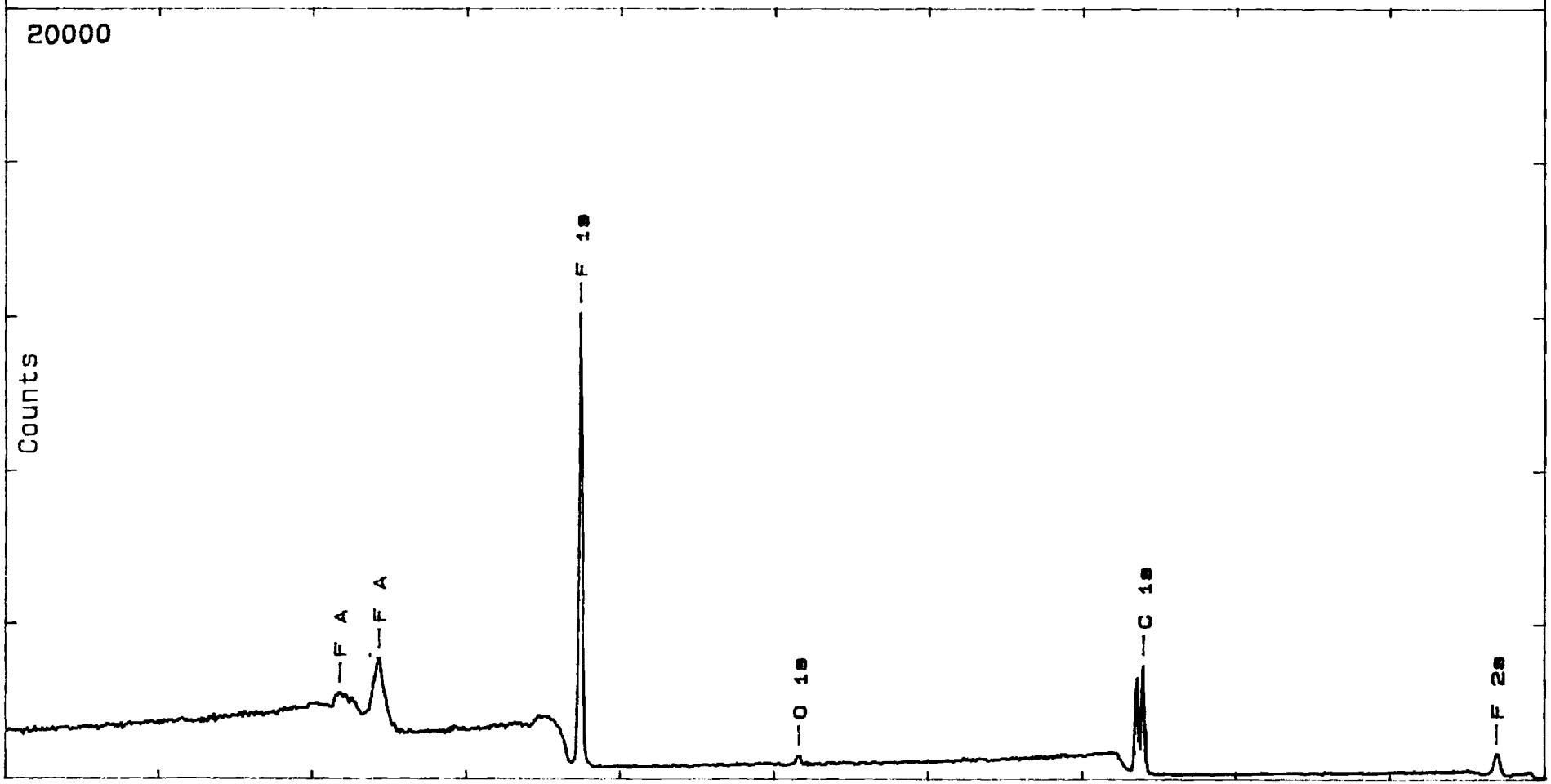


Report #: MSFC-01

File: PVDFMRS08	Date: 8/17/1988	Spot Size: 1000 u	Flood Gun: 7.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4

Description: PVDF-4, CONTROL (NO EXPOSURE)
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

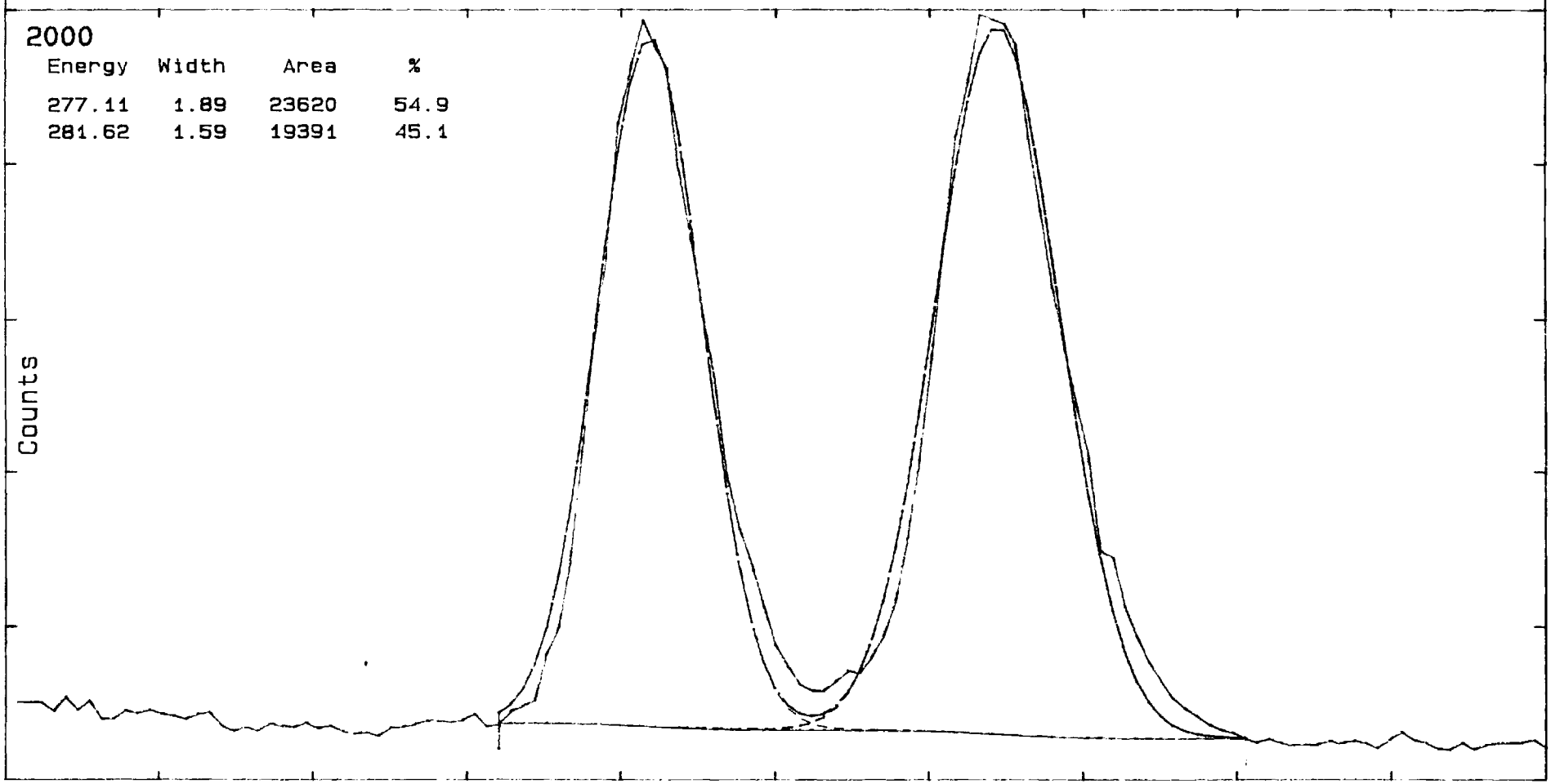


Report #: MSFC-01

File: PVDFMRS08	Date: 8/17/1988	Spot Size: 300 u	Flood Gun: 7.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-4 (CONTROL), C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

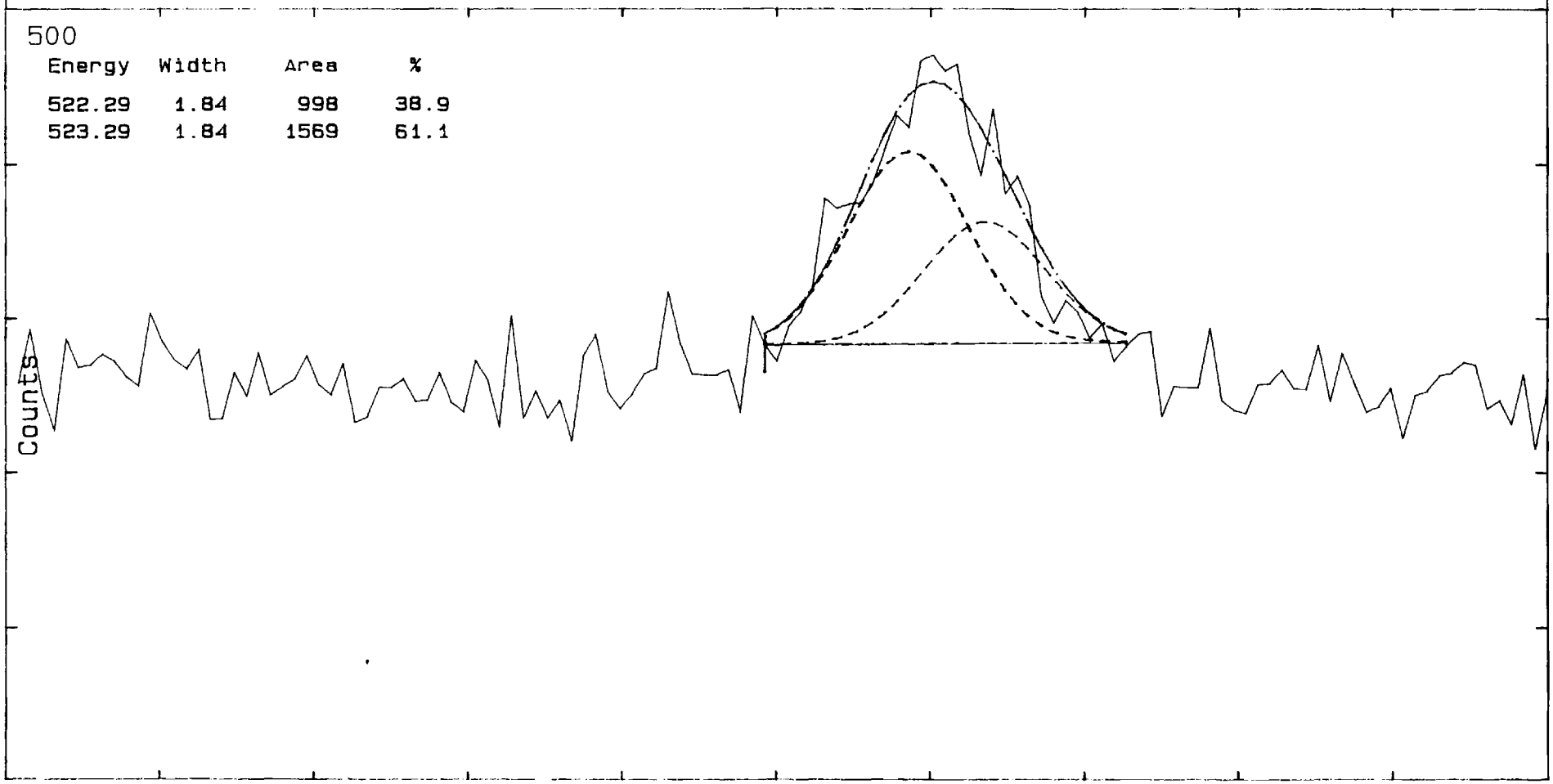


Report #: MSFC-01

File: PVDFMRS08	Date: 8/17/1988	Spot Size: 300 u	Flood Gun: 7.0 eV
Region 3	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-4 (CONTROL), O 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

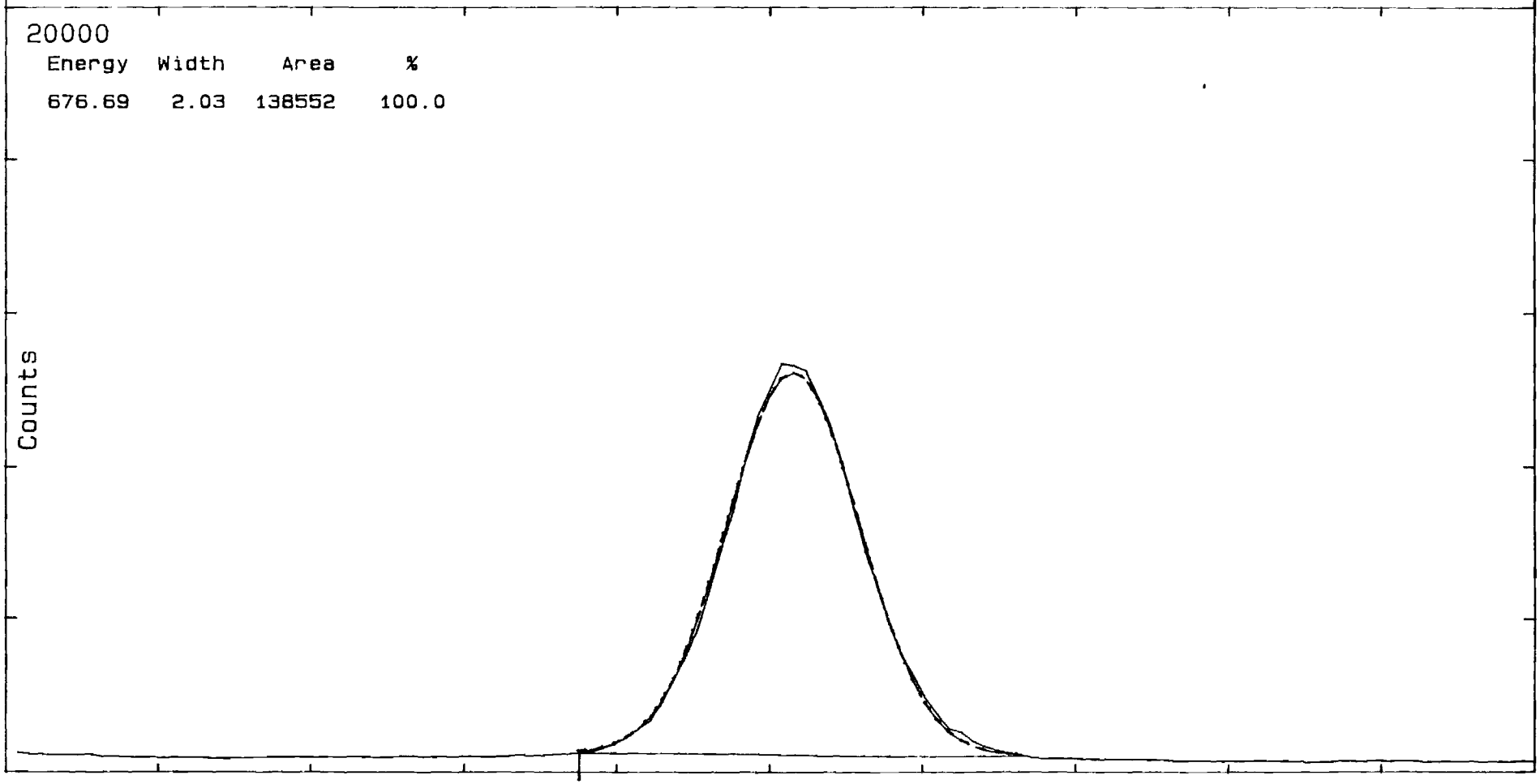


Report #: MSFC-01

File: PVDFMRS09	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 9.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-5, F 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

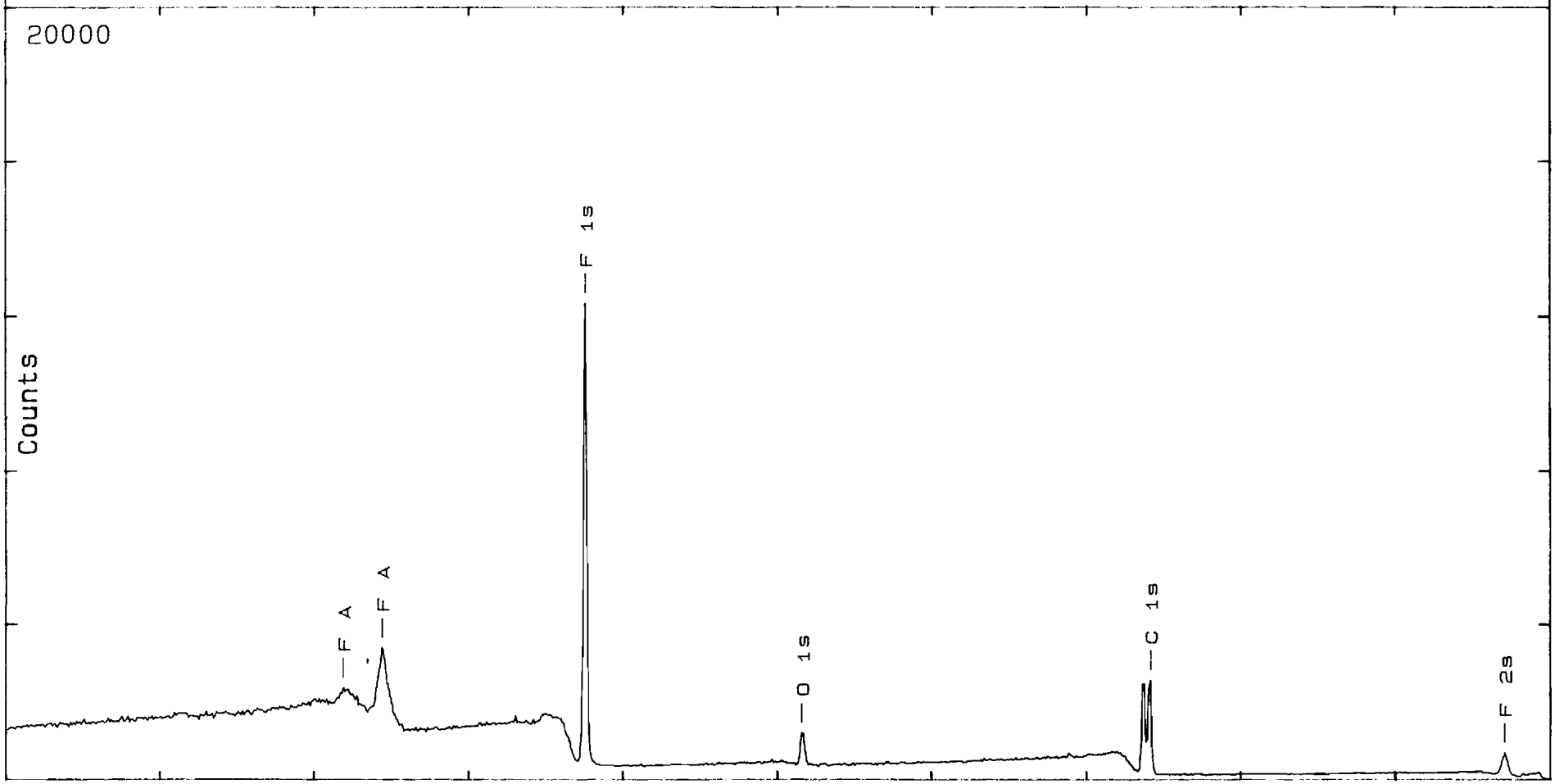


Report #: MSFC-01

File: PVDFMRS09	Date: 8/18/1988	Spot Size: 1000 u	Flood Gun: 9.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4

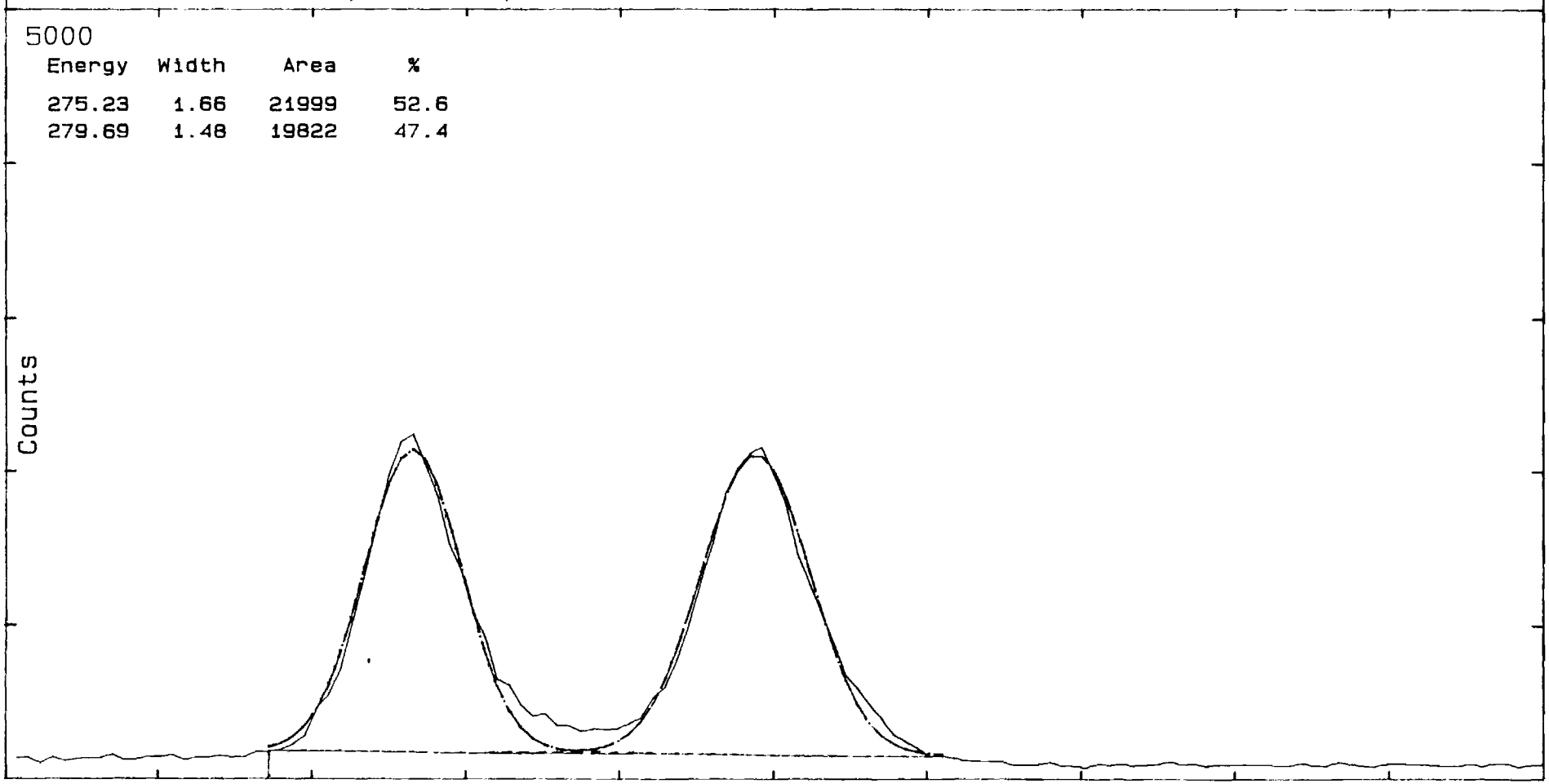
Description: PVDF-5, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN. CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

File: PVDFMRS09	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 9.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2
Description: PVDF-5, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN			Operator: WBC

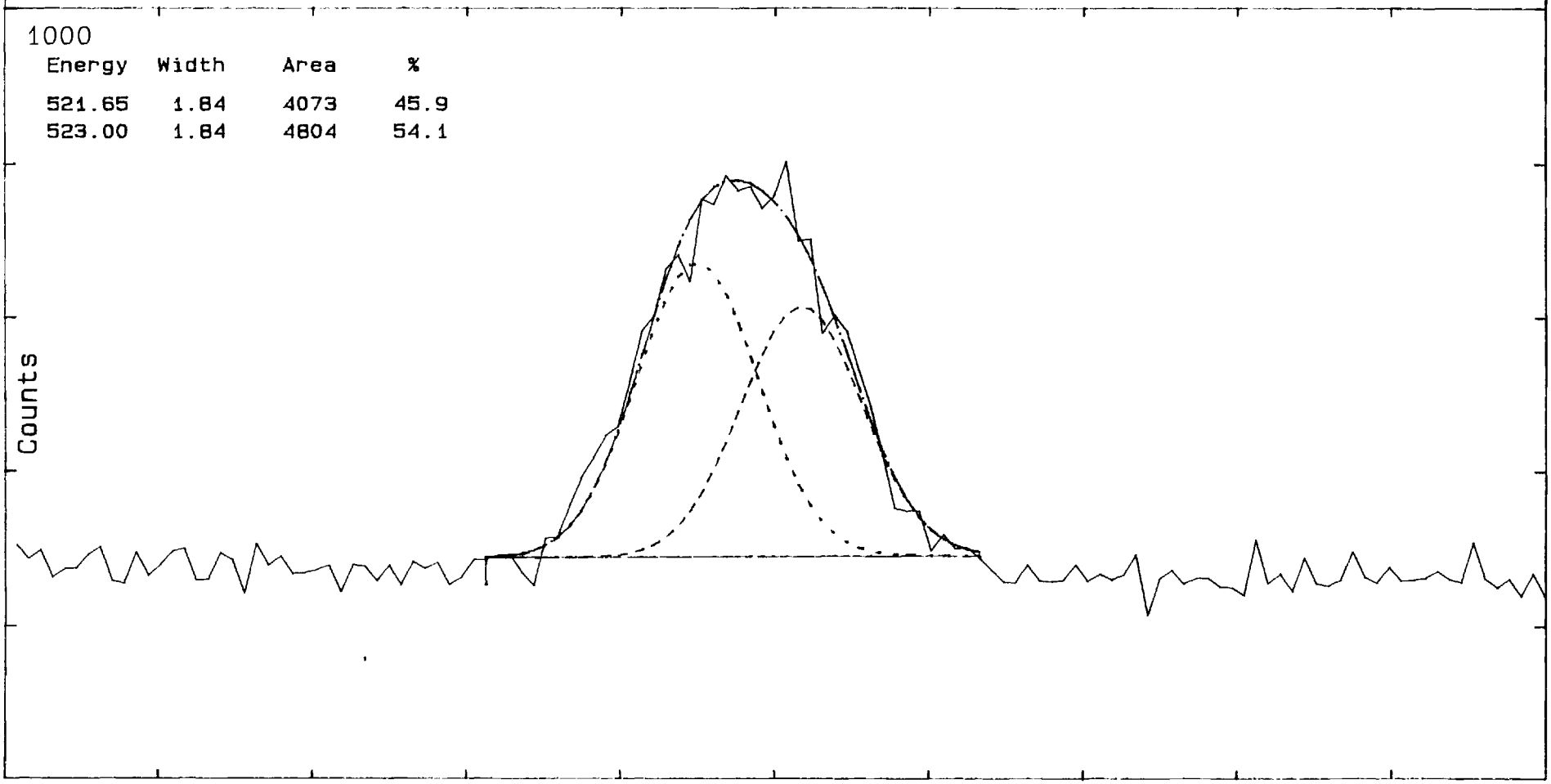


Report #: MSFC-01

File: PVDFMRS09	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 9.0 eV
Region 3	Disc: NASA01	# of Scans: 10	Resolution: 2

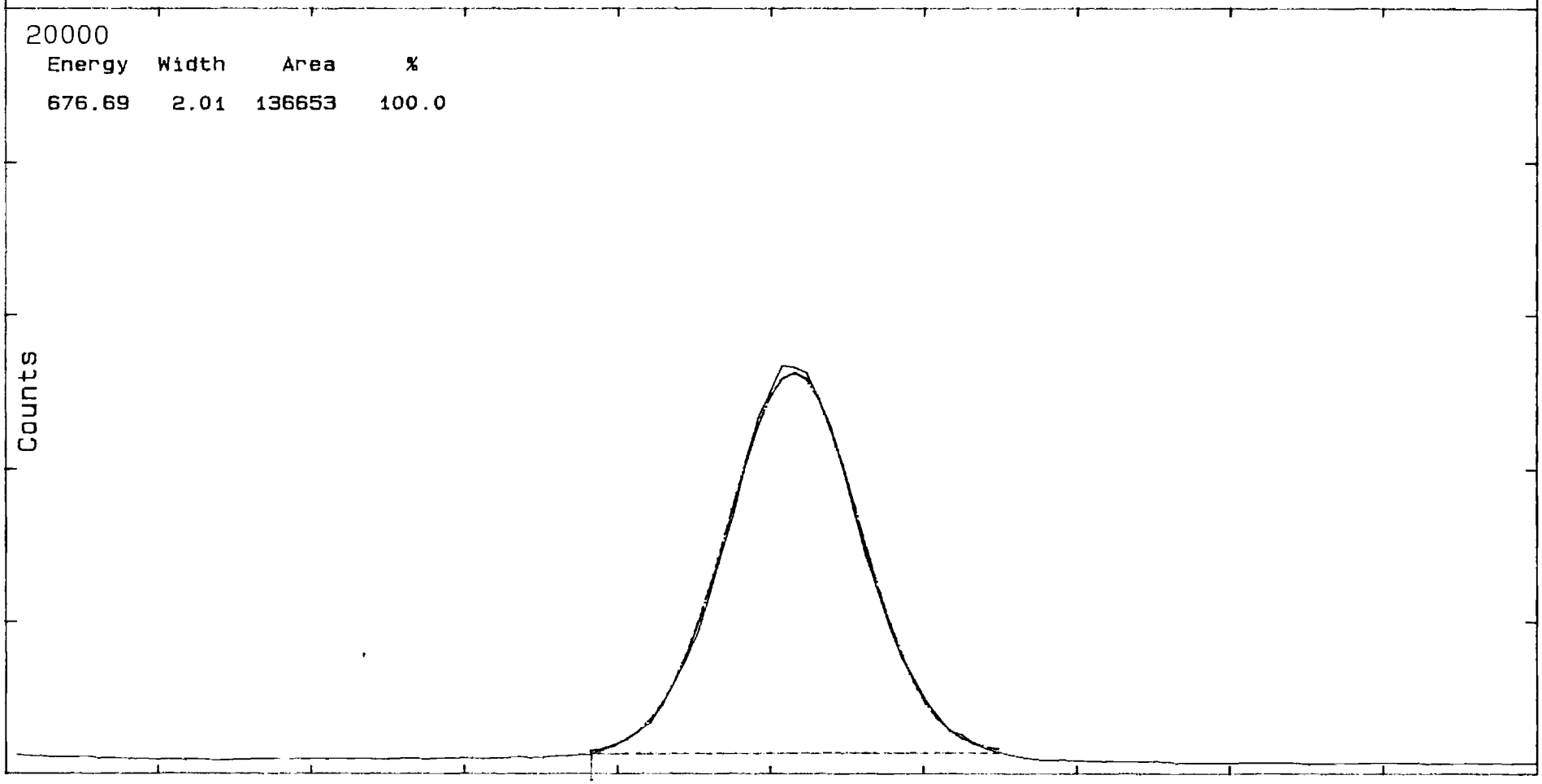
Description: PVDF-5, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC



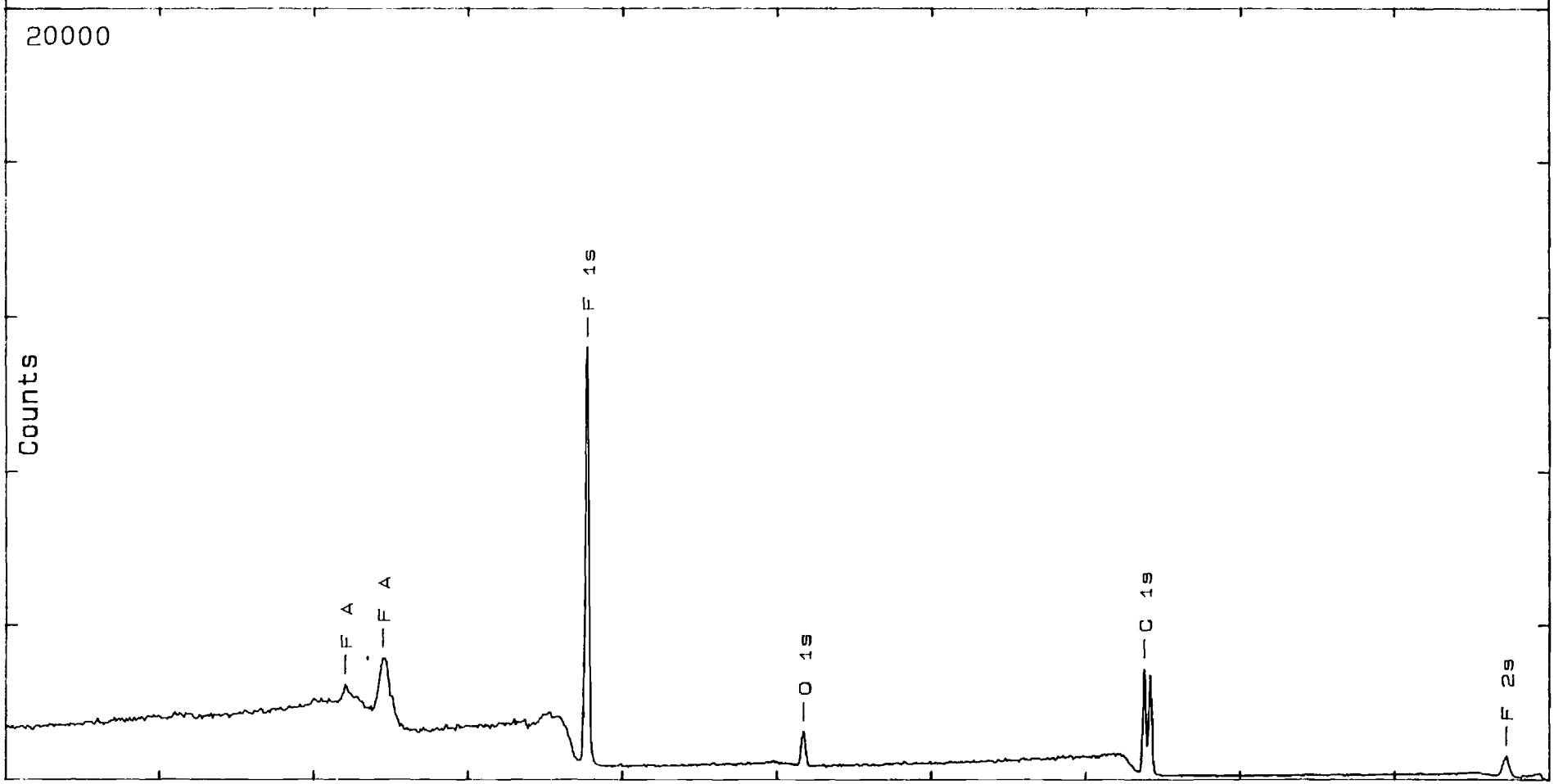
Report #: MSFC-01

File: PVDFMRS09	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 9.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2
Description: PVDF-5, F 1S SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN			Operator: WBC



Report #: MSFC-01

File: PVDFMRS10	Date: 8/18/1988	Spot Size: 1000 u	Flood Gun: 11.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4
Description: PVDF-6, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN. CHARGE NEUTRALIZATION SCREEN			Operator: WBC

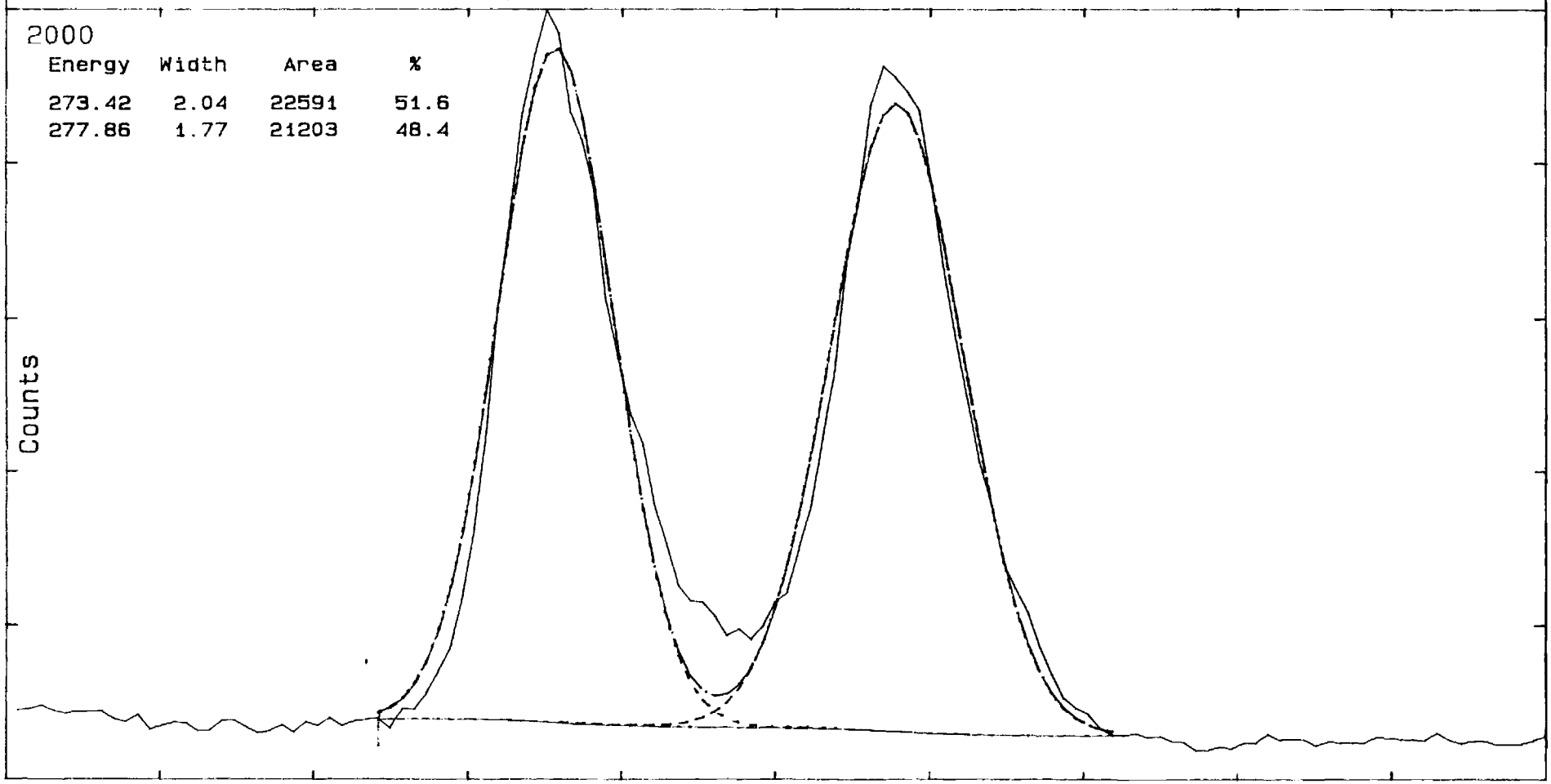


Report #: MSFC-01

File: PVDFMRS10	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 11.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-6, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

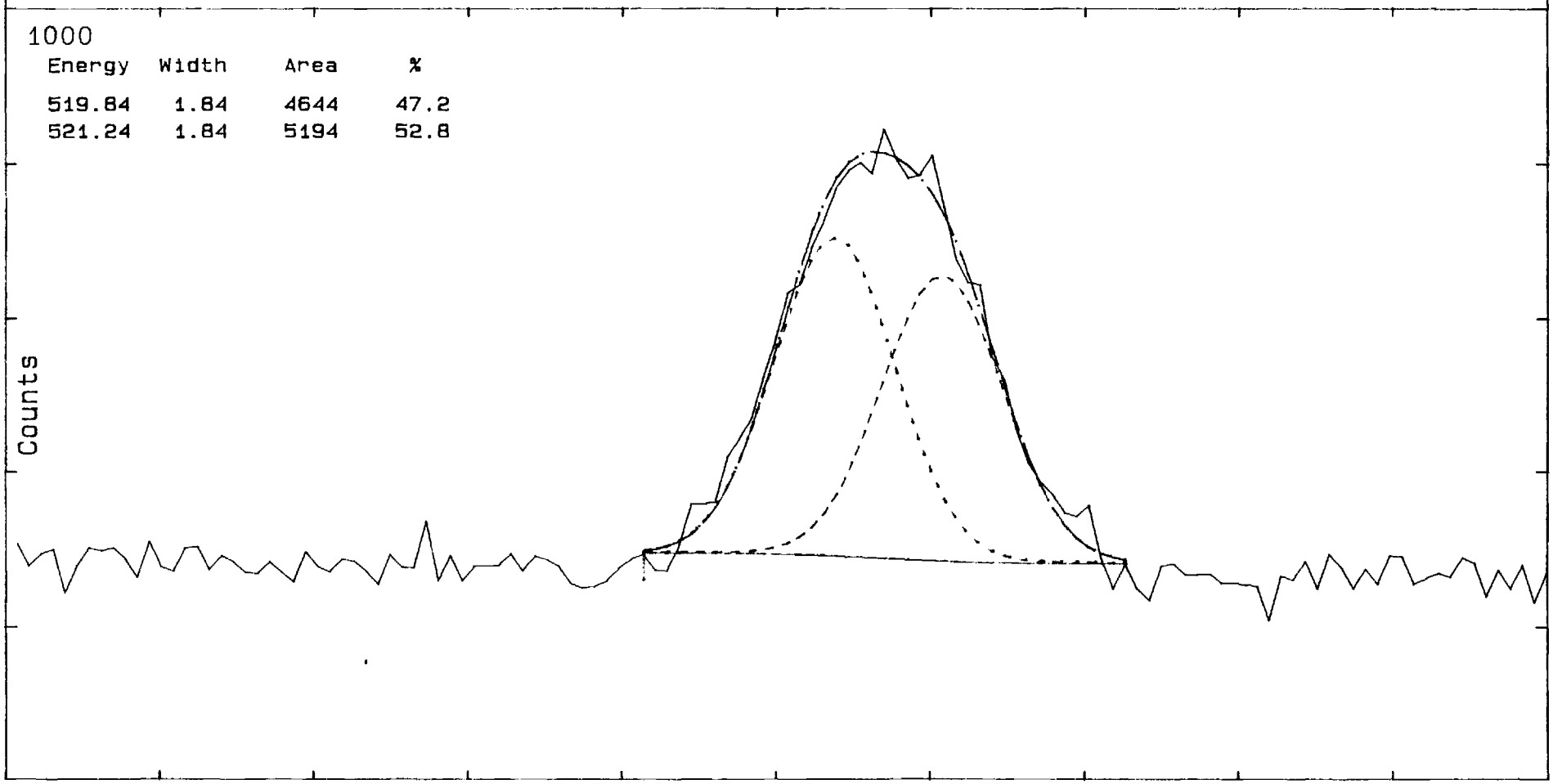


Report #: MSFC-01

File: PVDFMRS10	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 11.0 eV
Region 3	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-6, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

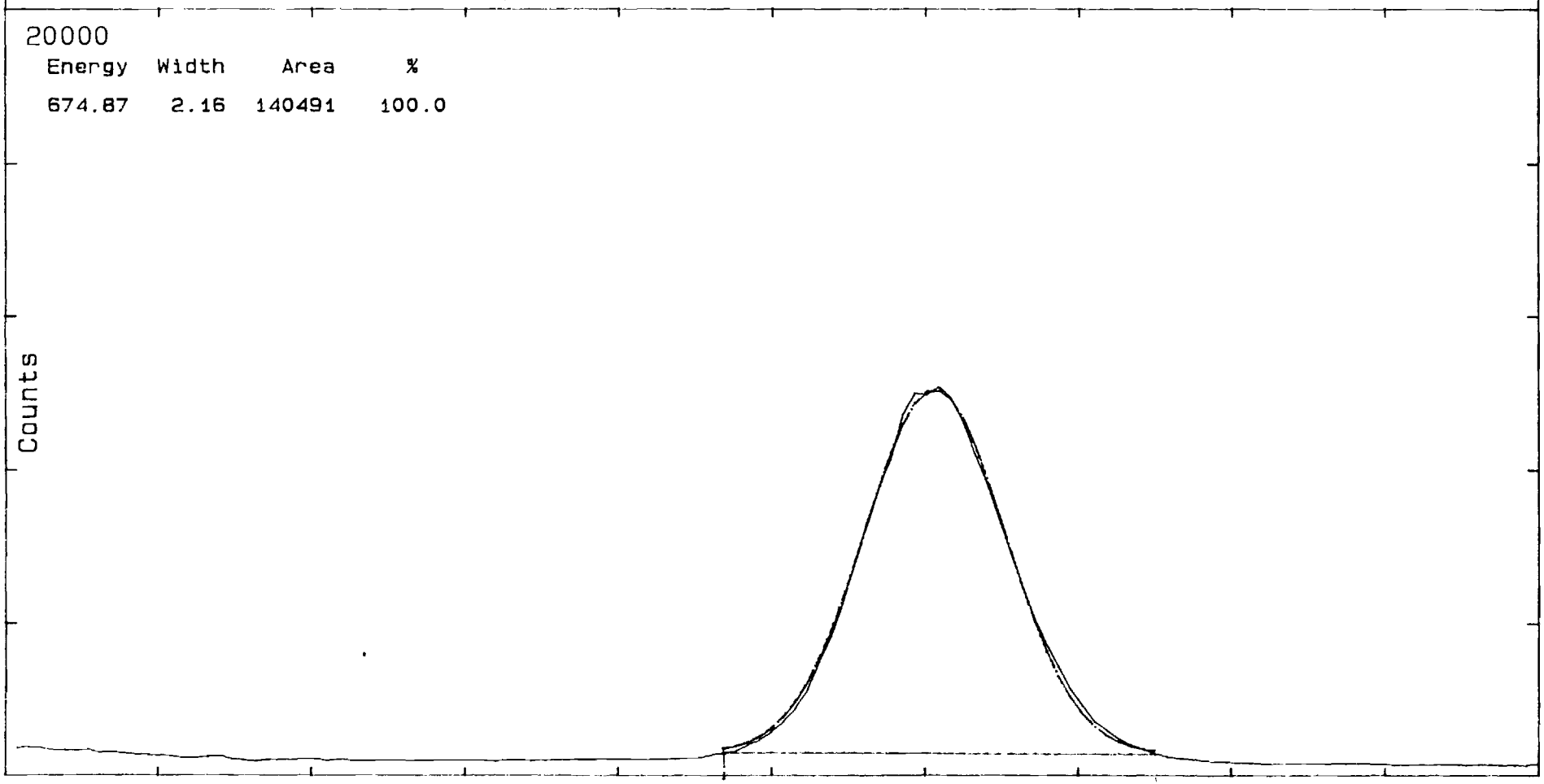


Report #: MSFC-01

File: PVDFMRS10	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 11.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-6, F 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

CONTRACTOR:	Georgia Tech Research Corporation Georgia Institute of Technology Atlanta, Georgia 30332-0420
REPORT NUMBER:	MSFC-02
TITLE OF REPORT:	Electron Spectroscopy for Chemical Analysis - Sample Analysis
DATE OF PUBLICATION:	29 August 1988
TYPE OF REPORT:	Sample Analysis
REQUISITION NUMBER:	1-7-EH-91814(1F)
AUTHOR:	W. B. Carter

PREPARED FOR:

George C. Marshall Space Flight Center
Marshall Space Flight Center, AL 35812

I. INTRODUCTION

This report describes the results of the ESCA analysis of the specimens listed in Table 1, which were supplied by MSFC. ESCA data collection and reduction were performed as described in Report # MSFC-01 dated 23 August 1988.

TABLE 1

Specimens Analyzed with ESCA

Specimen	Exposure Conditions in Atomic Oxygen
MYLAR CONTROL	Control (no exposure)
MYLAR-11	Exposed at 45°C, 60 W, 75 Min.
POLYPROPYLENE CONTROL	Control (no exposure)
POLYPROPYLENE-11	Exposed at 45°C, 100 W, 90 Min.

II. DATA ANALYSIS

A. Compositional Analysis.

The spectra collected are presented in the appendix. Table 2 lists the composition of the surface of each specimen, with the exception of specimen Mylar-11, as determined semiquantitatively from its general survey. The surface of Mylar-11 has a substantial amount of Sb on it. Since the Sb 3d5/2 photoionization peak overlaps the O 1s photoionization peak, quantification was not possible. The Mylar control specimen may have a small amount of Sb on it (see below), which has been ignored in the quantification. The atomic percents are given to the nearest percent and thus may not sum to 100%.

B. Analysis of Specimens: MYLAR AND MYLAR-11.

The general survey from the control specimen (MYLAR) indicates that the specimen surface is composed primarily of C and O, with a trace of F and possibly Sb contamination. The C 1s spectrum of this specimen resolves into five components.

TABLE 2

Surface Composition of Polymer Specimens

Specimen	Atomic Concentration (%) C	O	F	Al	Na
MYLAR	75	24	0.5	-	-
MYLAR-11*					
POLYPROPYLENE	97	3	-	-	-
POLYPROPYLENE-11	80	14	2.5	3	0.5

* The surface of MYLAR-11 consists primarily O and C, with smaller amounts of Sb, Ca, Na, N, and P.

The O 1s spectrum of the unexposed specimen resolves into two components.

The general survey spectra of the plasma exposed specimen (MYLAR-11) indicates an increased O to C ratio relative to the unexposed specimen. The C 1s spectrum of this specimen differs from that of the unexposed specimen. Assuming that the C 1s peak that appears at 282.1 eV on the control specimen is due to C in the same state as that which produces the peak at 273.9 eV on the exposed specimen, the average C 1s binding energy is greater on the exposed specimen than on the control. The lack of an absolute binding energy reference (see Report # MSFC-01) prevents a more conclusive analysis at this time.

The O 1s spectrum of the plasma exposed specimen overlaps the Sb 3d5/2 peak. Peak fitting was not attempted. The Sb 3d3/2 peak can be seen at a binding energy of about 529.5 eV.

C. Analysis of Specimens: POLYPROPYLENE and POLYPROPYLENE-11.

The general survey of the control specimen (POLYPROPYLENE) indicates that the specimen surface is composed primarily of C and O. As there should be no O in the pure polymer, the O may be due to a slight oxidation of the surface and/or contamination. The solvent used to clean the specimen may have been the source of the O observed.

The C1s spectrum of this specimen resolves into two primary peaks due to the different bonding states of C in the polymer and a small peak at a greater binding energy. This small component may be the result of surface oxidation.

The O 1s spectrum of the control specimen resolves into two components.

The general survey spectrum of the plasma exposed specimen (POLYPROPYLENE-11) indicates an increased O to C ratio relative to the unexposed specimen. Several impurities also are present on the plasma exposed specimen, which are not seen on the control.

The C 1s spectrum of the plasma exposed specimen is broader than that from the unexposed specimen and resolves into three primary components and one or more components at greater binding energies. This structure is consistent with oxidation of the polymer.

The O 1s peak of the plasma exposed specimen resolves into two components.

III. SUMMARY

Plasma treatment appears to oxidize both of the polymers examined. Evidence for this are the increases in O on the specimen surfaces after plasma exposure and the increases in the relative amounts of C in various oxidized states following exposure.

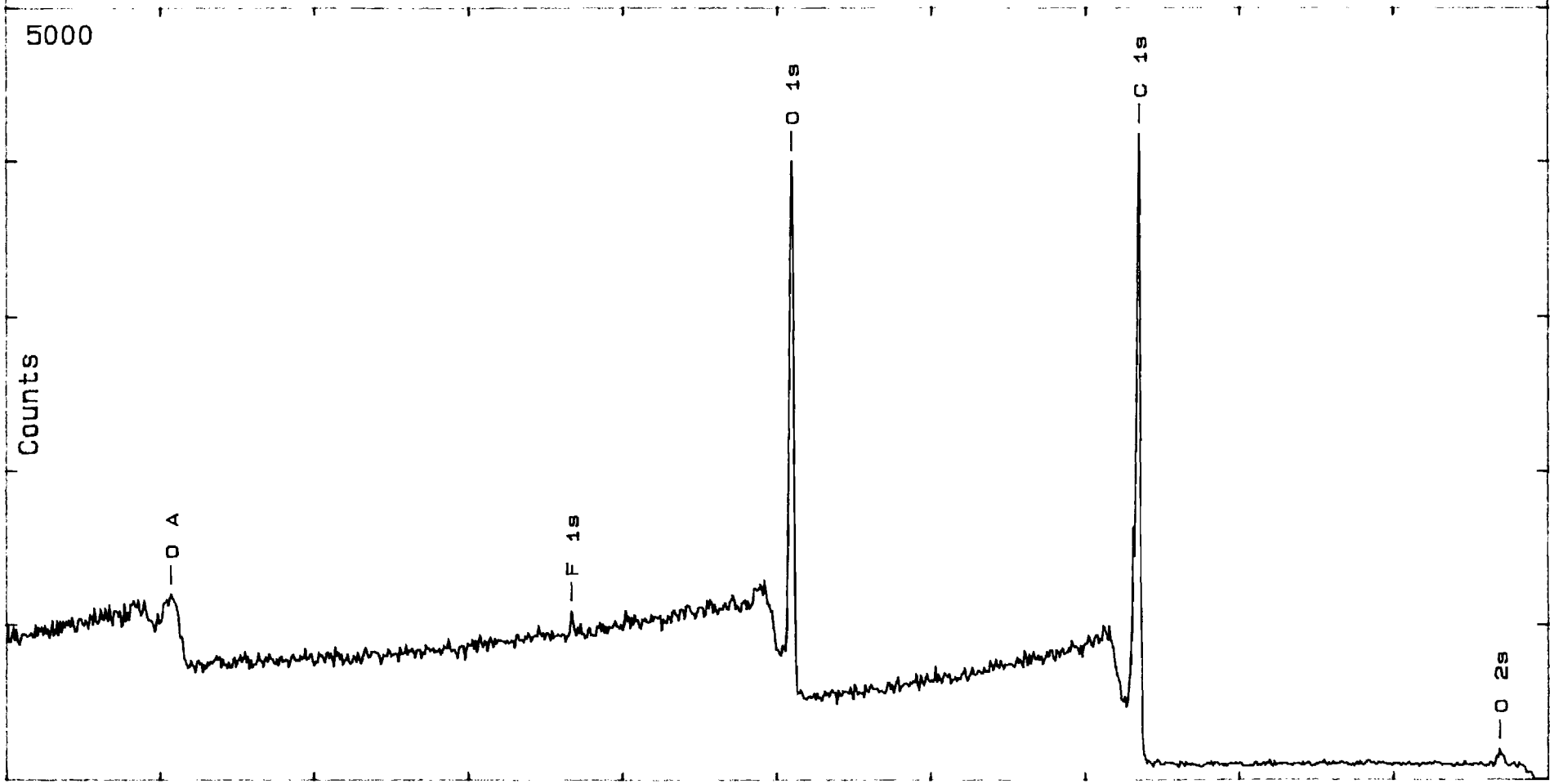
It is not possible to quantify the relative extent of the oxidation observed because of the difficulty imposed by the presence of Sb on MYLAR-11 (see above). In any case, this would most likely be a meaningless exercise since the specimens were exposed in different systems under different conditions.

The surfaces of both plasma exposed specimens contained impurities. This may be indicative of contamination in the plasma reactors.

APPENDIX

ESCA SPECTRA

File: MYLAR01	Date: 8/25/1988	Spot Size: 1000 u	Flood Gun: 1.0 eV
	Disc: NASA02	# of Scans: 1	Resolution: 4
Description: MYLAR CONTROL CHARGE NEUTRALIZATION SCREEN			Operator: WBC

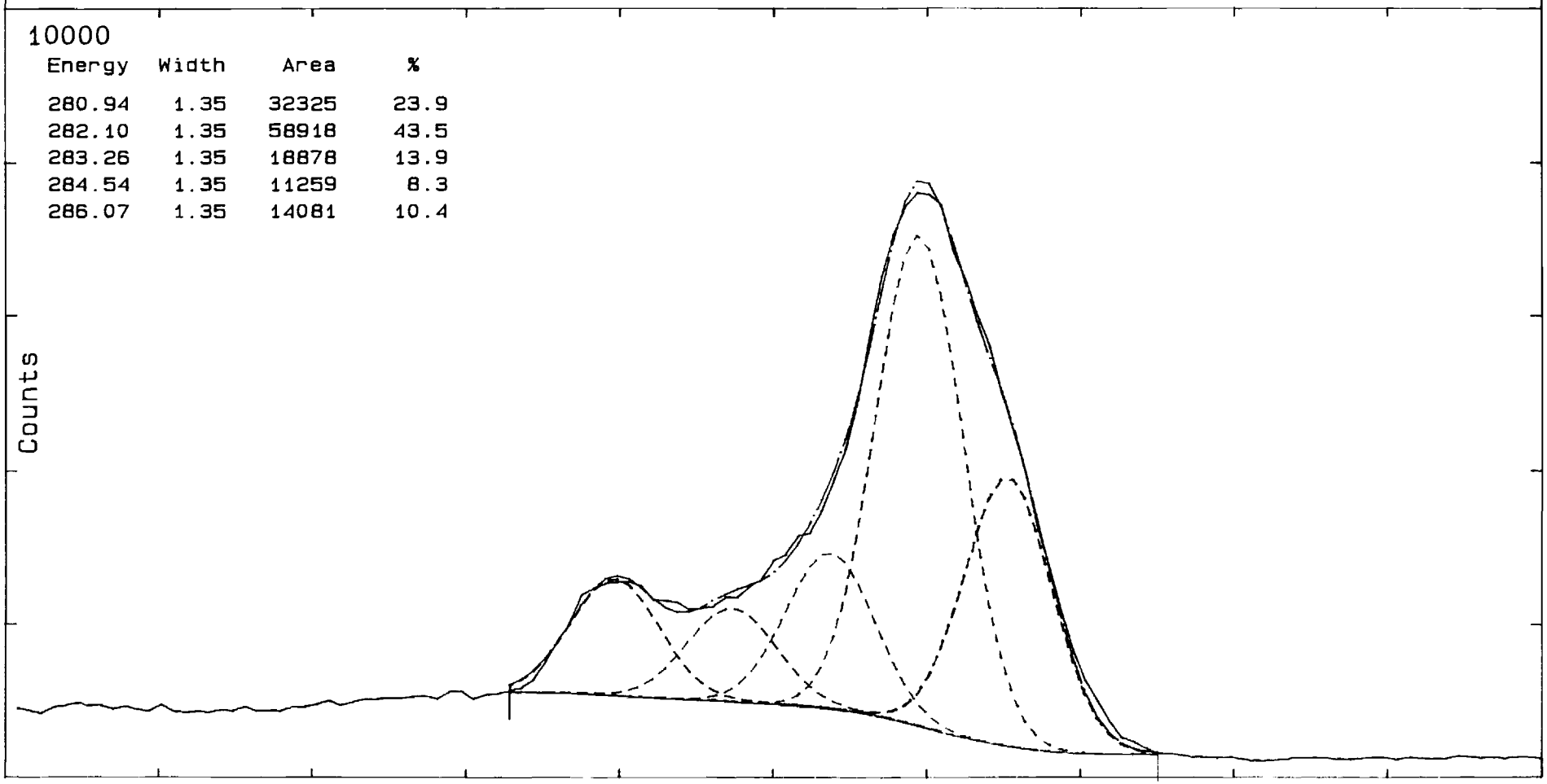


Report #: MSFC-02

File: MYLARMRS01	Date: 8/25/1988	Spot Size: 300 u	Flood Gun: 1.0 eV
Region 1	Disc: NASA02	# of Scans: 30	Resolution: 2

Description: MYLAR CONTROL, C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

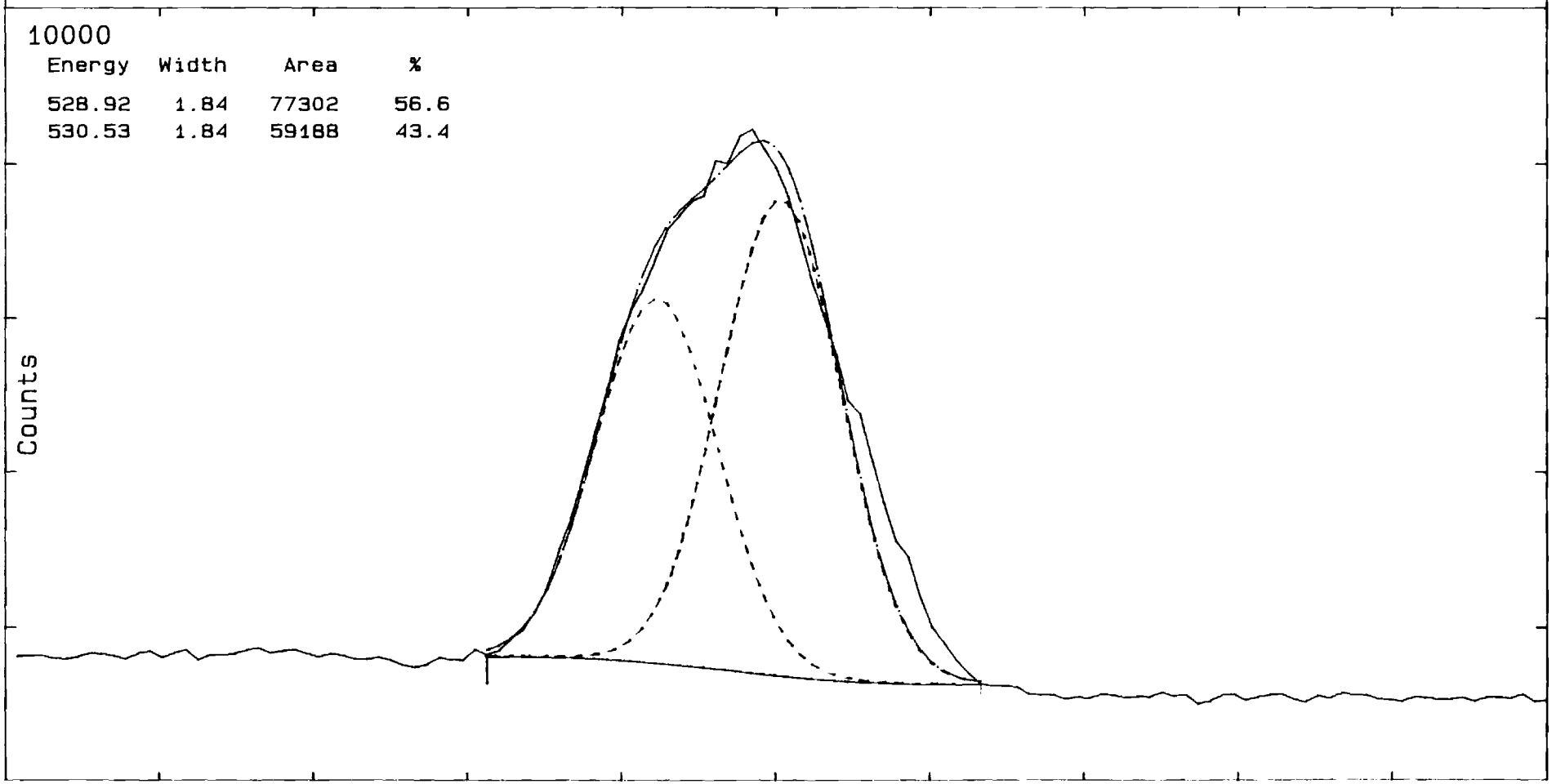


Report #: MSFC-02

File: MYLARMRS01	Date: 8/25/1988	Spot Size: 300 u	Flood Gun: 1.0 eV
Region 2	Disc: NASA02	# of Scans: 30	Resolution: 2

Description: MYLAR CONTROL, O 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

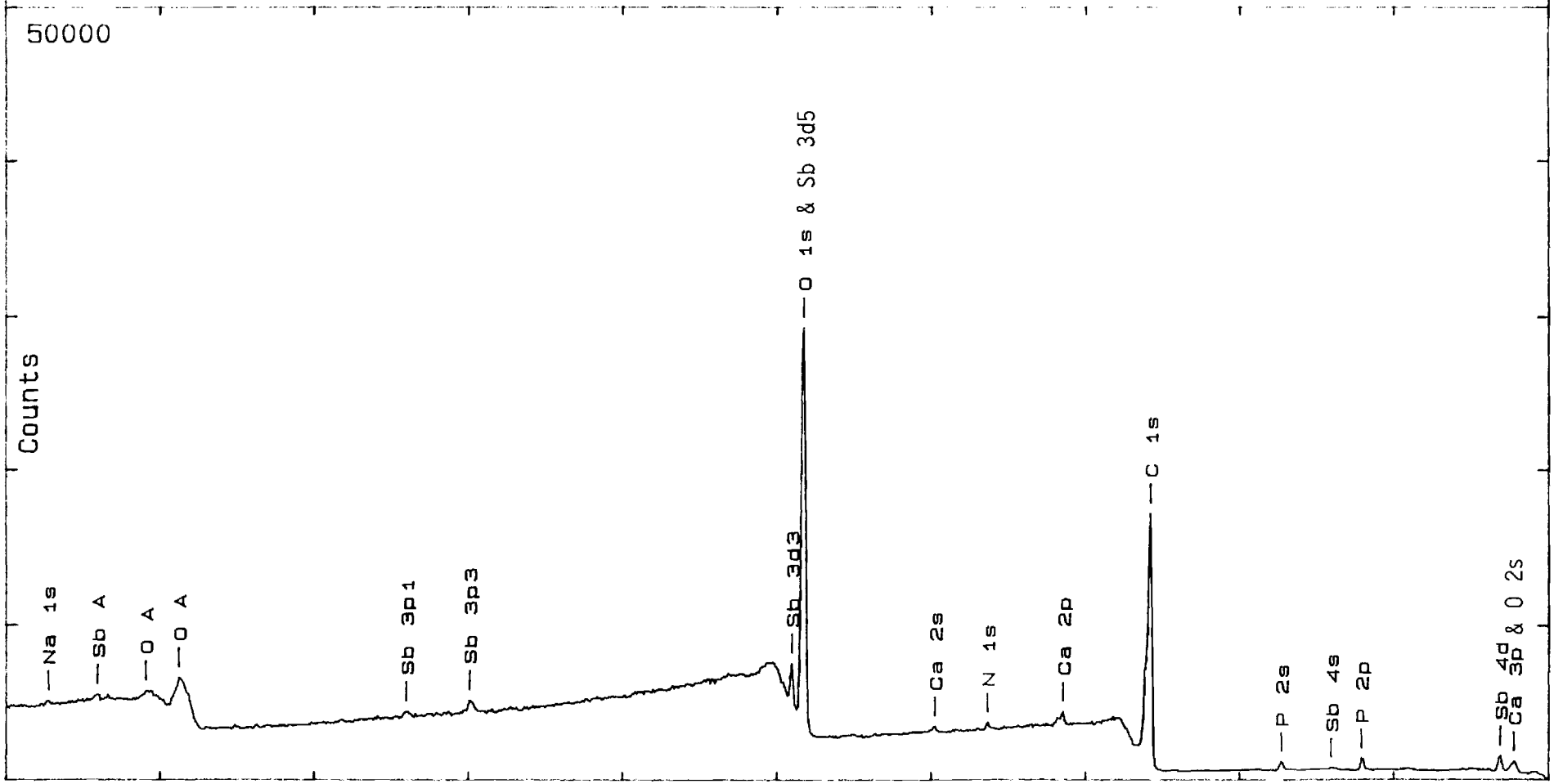


Report #: MSFC-02

File: MYLAR02	Date: 8/25/1988	Spot Size: 1000 u	Flood Gun: 9.0 eV
	Disc: NASA02	# of Scans: 5	Resolution: 4

Description: MYLAR-11, PLASMA EXPOSED: 45 DEG C, 60 W, 75 MIN.
CHARGE NEUTRALIZATION SCREEN

Operator: WBC



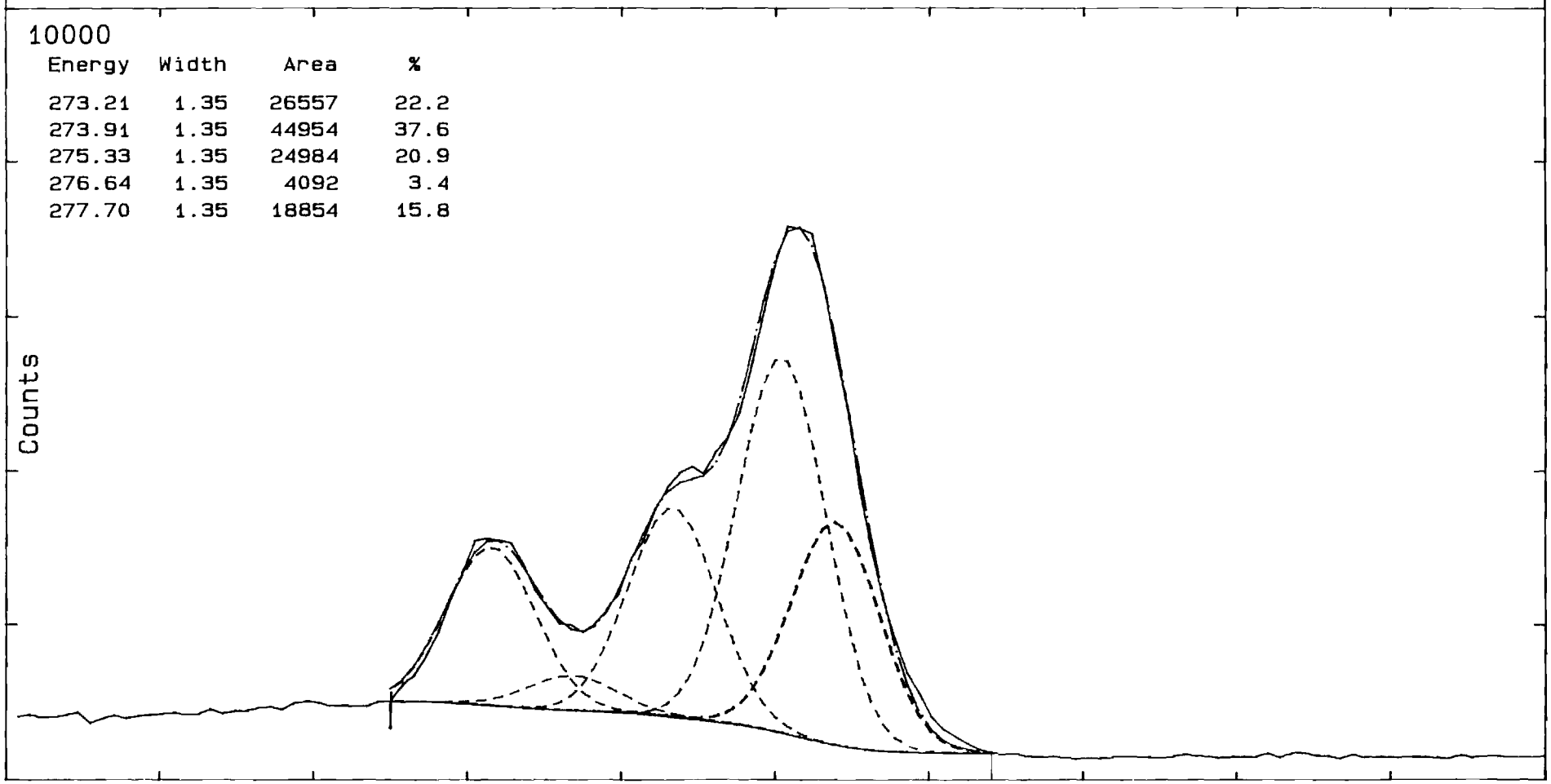
1090.0 Binding Energy (eV) -10.0

Report #: MSFC-02

File: MYLARMRS03	Date: 8/25/1988	Spot Size: 300 u	Flood Gun: 9.0 eV
Region 1	Disc: NASA02	# of Scans: 30	Resolution: 2

Description: MYLAR-11, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 75 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

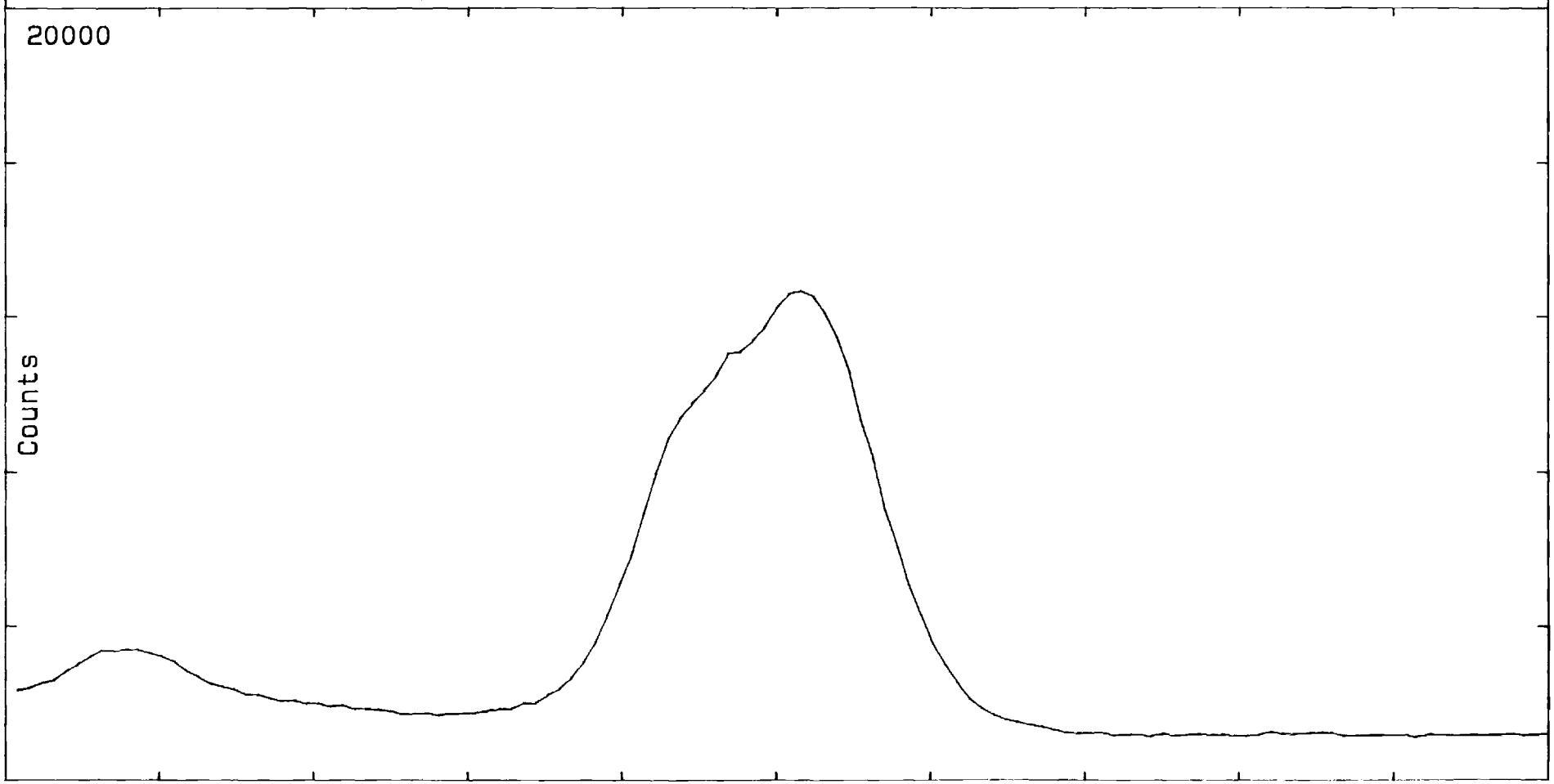


Report #: MSFC-02

File: MYLARMRS03	Date: 8/25/1988	Spot Size: 300 u	Flood Gun: 9.0 eV
Region 2	Disc: NASA02	# of Scans: 30	Resolution: 2

Description: MYLAR-11, O 1s & Sb 3d SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 75 MIN., NEUTRALIZATION SCREEN

Operator: WBC

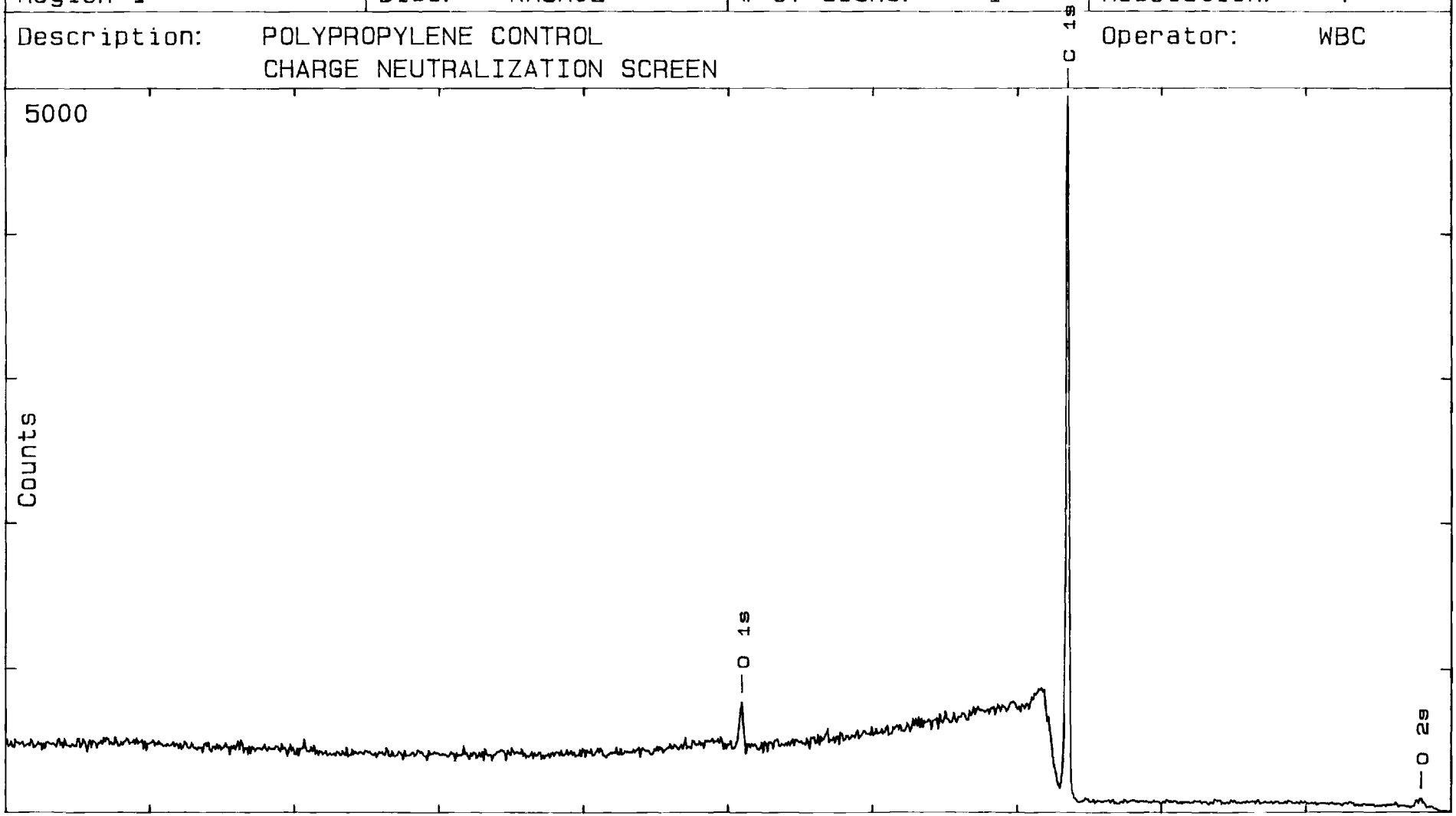


Report #: MSFC-02

File: PPROPMRS01	Date: 8/26/1988	Spot Size: 1000 u	Flood Gun: On, 0 eV
Region 1	Disc: NASA02	# of Scans: 1	Resolution: 4

Description: POLYPROPYLENE CONTROL
CHARGE NEUTRALIZATION SCREEN

Operator: WBC



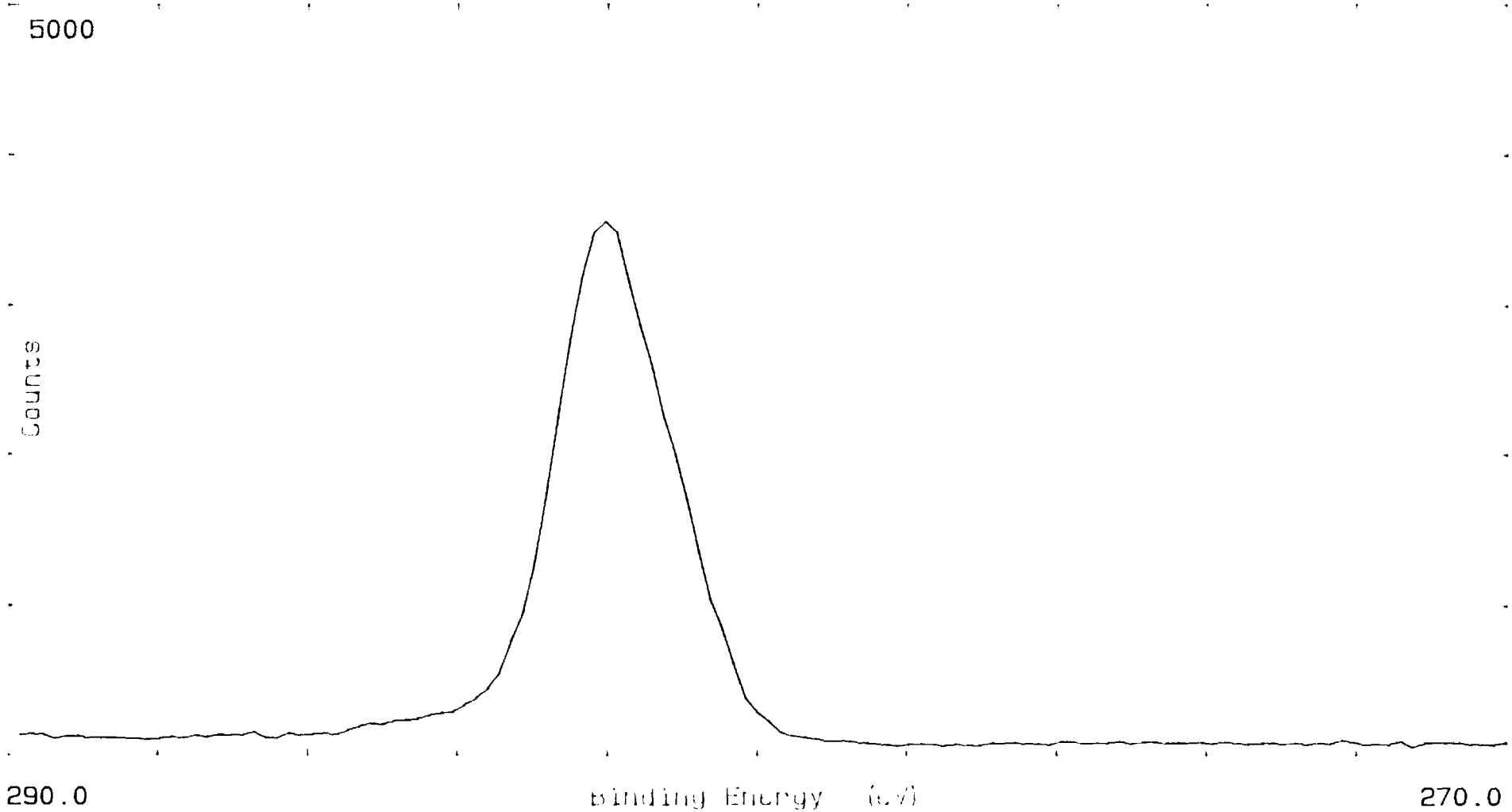
1090.0 Binding Energy (eV) -10.0

Report #: MSFC-02

File: PPROPMRS01	Date: 8/26/1988	Spot Size: 300 u	Flood Gun: On, 0 eV
Region 2	Disc: NASA02	# of Scans: 10	Resolution: 2

Description: POLYPROPYLENE CONTROL, C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

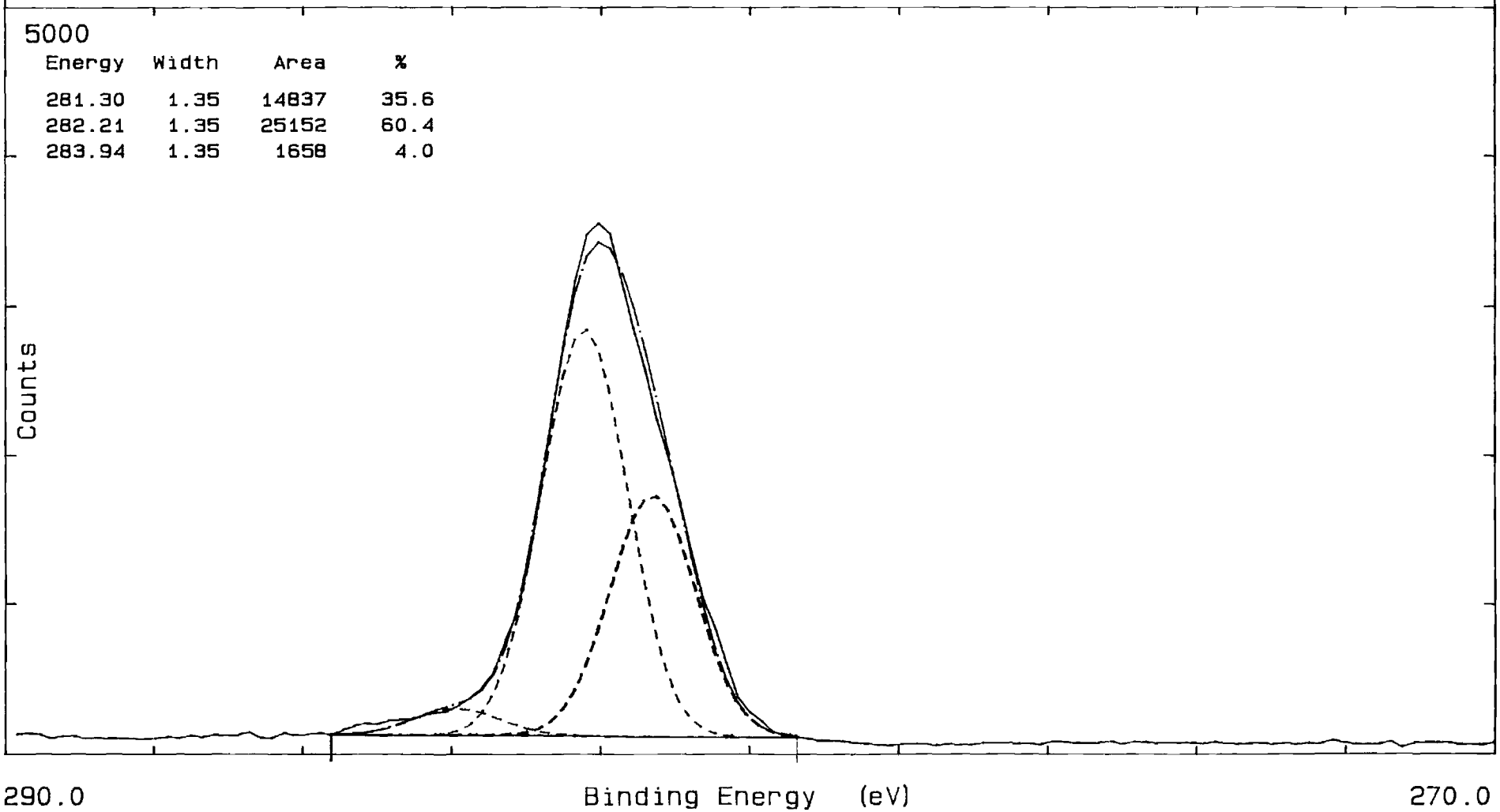


Report #: MSFC-02

File: PPROPMRS01	Date: 8/26/1988	Spot Size: 300 u	Flood Gun: On, 0 eV
Region 2	Disc: NASA02	# of Scans: 10	Resolution: 2

Description: POLYPROPYLENE CONTROL, C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

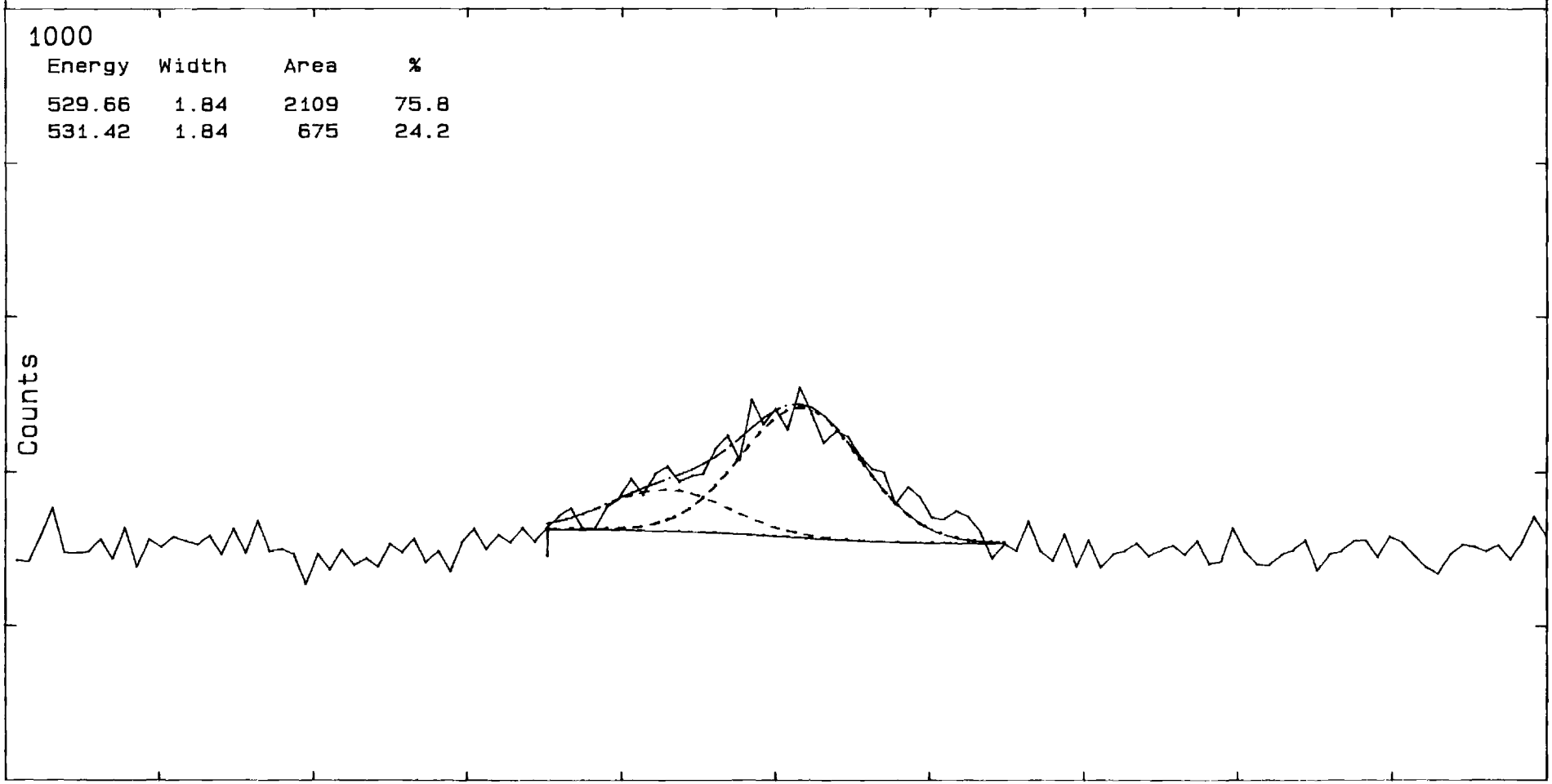


Report #: MSFC-02

File: PPROPMRS01	Date: 8/26/1988	Spot Size: 300 u	Flood Gun: On, 0 eV
Region 3	Disc: NASA02	# of Scans: 10	Resolution: 2

Description: POLYPROPYLENE CONTROL, O 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

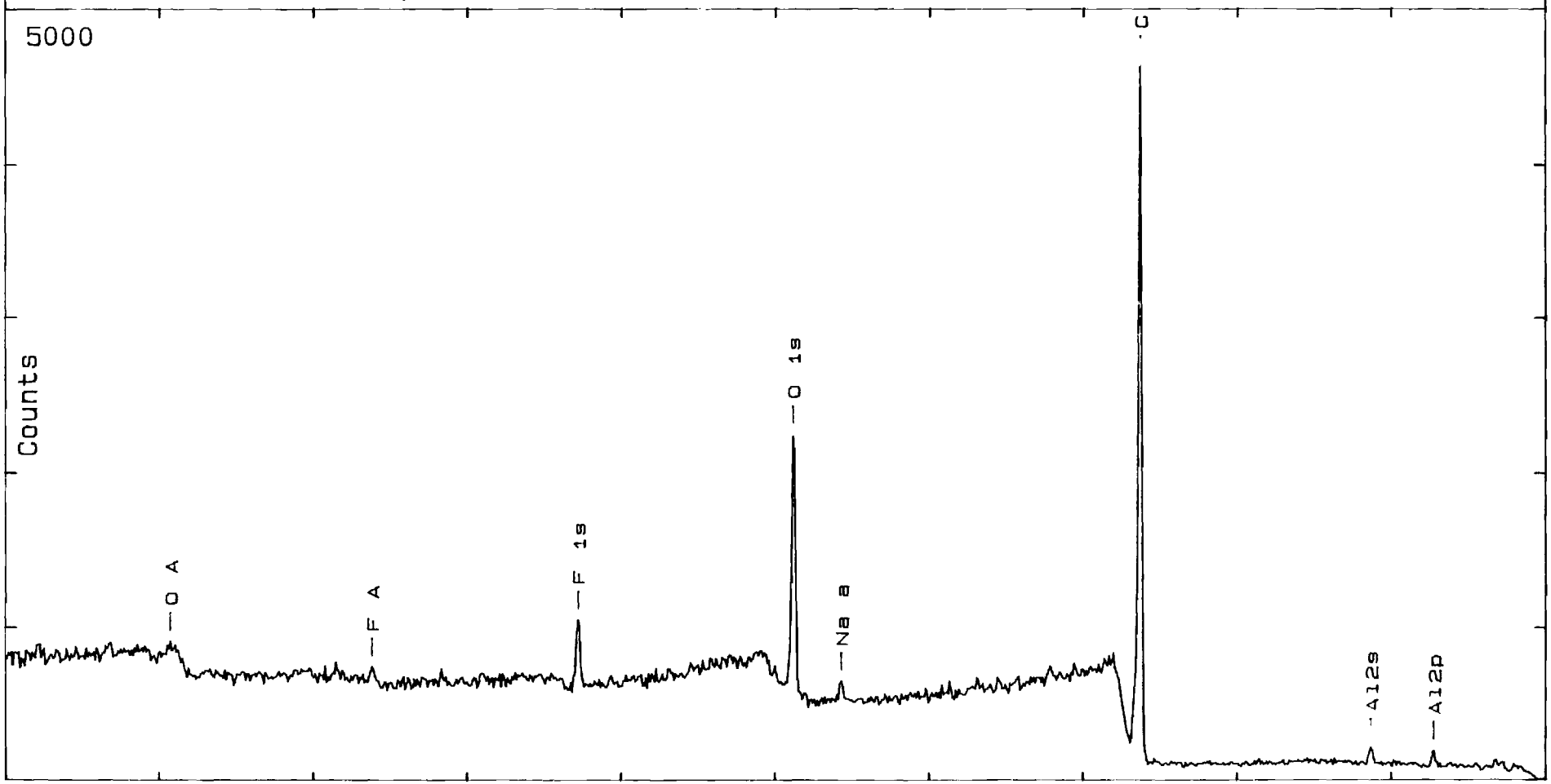


Report #: MSFC-02

File: PPROPMRS02	Date: 8/26/1988	Spot Size: 1000 u	Flood Gun: 3.0 eV
Region 1	Disc: NASA02	# of Scans: 1	Resolution: 4

Description: POLYPROPYLENE-11, PLASMA EXPOSED: 45 DEG C, 100 W, 90 MIN., CHARGE NEUTRALIZATION SCREEN

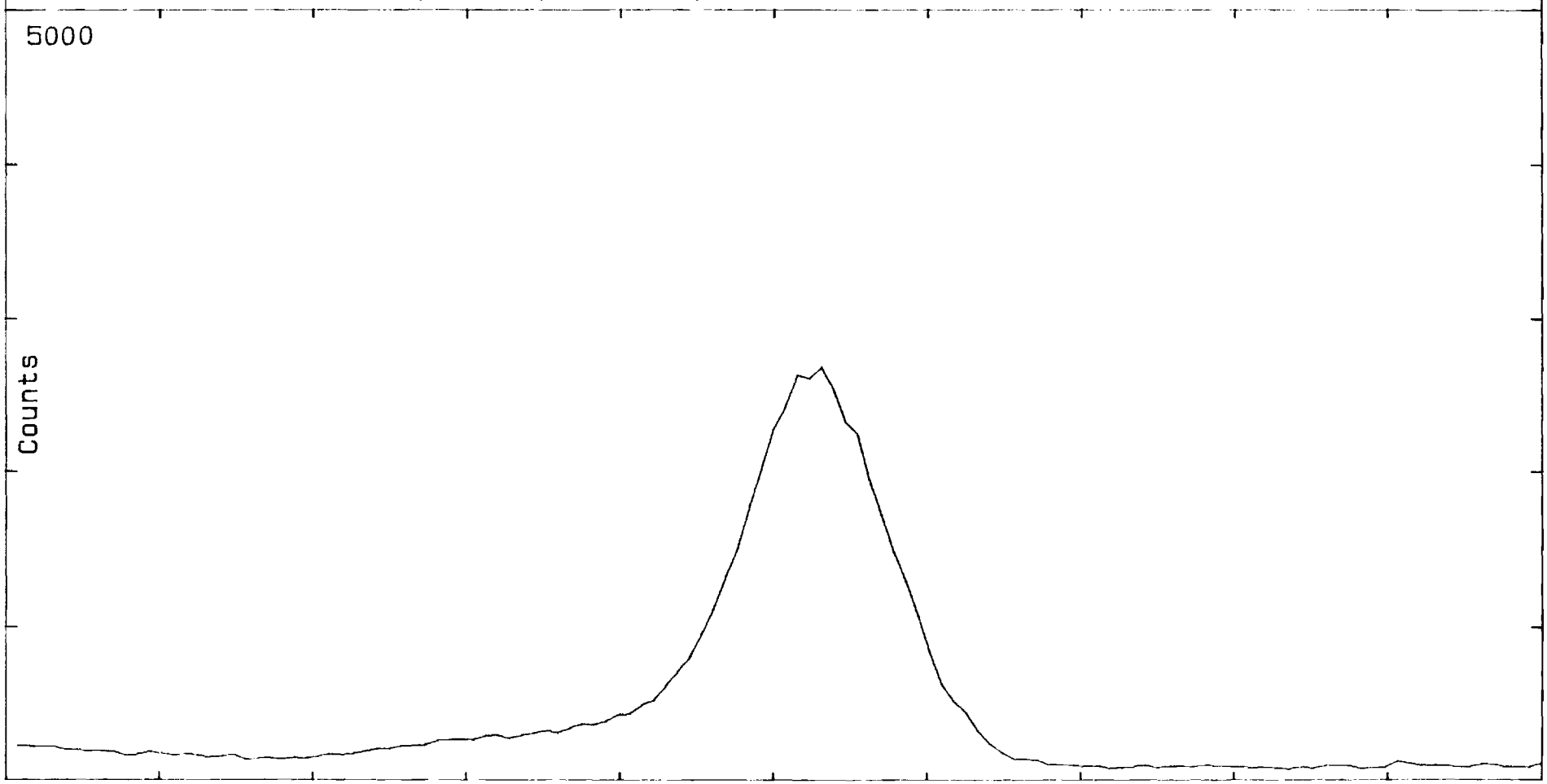
Operator: WBC



1090.0 Binding Energy (eV) -10.0

Report #: MSFC-02

File: PPROPMRS02	Date: 8/26/1988	Spot Size: 300 u	Flood Gun: 3.0 eV
Region 2	Disc: NASA02	# of Scans: 10	Resolution: 2
Description: POLYPROPYLENE-11, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 100 W, 90 MIN., NEUTRALIZATION SCREEN			Operator: WBC

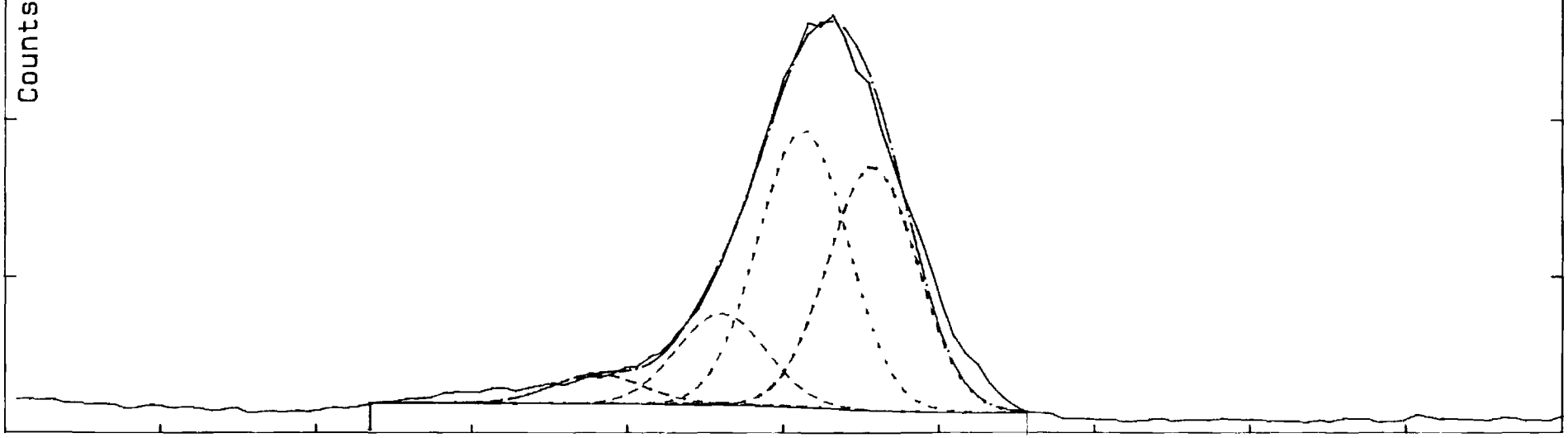


Report #: MSFC-02

File: PPROPMRS02	Date: 8/26/1988	Spot Size: 300 u	Flood Gun: 3.0 eV
Region 2	Disc: NASA02	# of Scans: 10	Resolution: 2
Description: POLYPROPYLENE-11, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 100 W, 90 MIN., NEUTRALIZATION SCREEN			Operator: WBC

5000

Energy	Width	Area	%
278.85	1.35	14349	38.0
279.75	1.35	16322	43.2
280.79	1.35	5393	14.3
282.40	1.35	1712	4.5



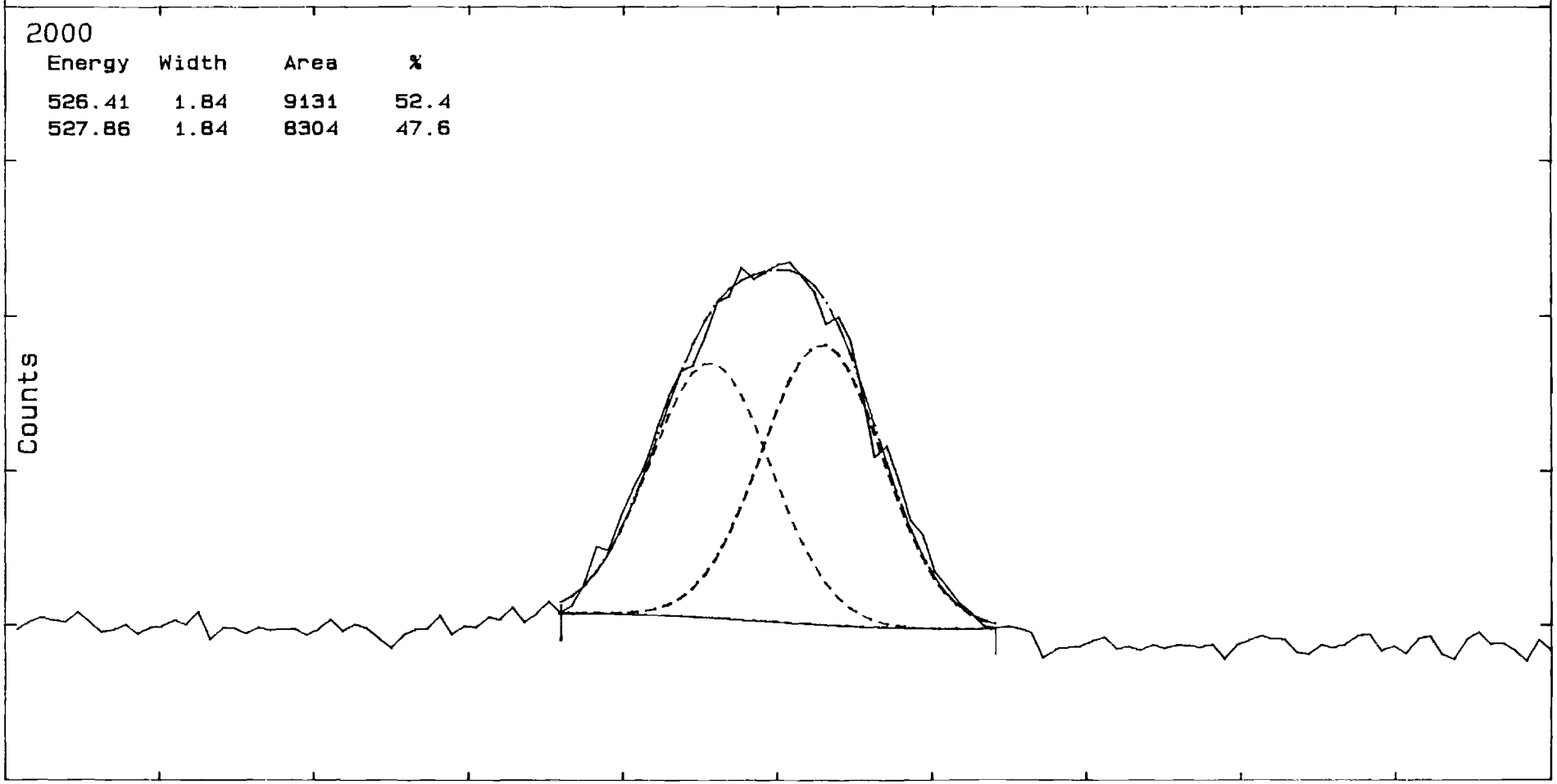
290.0 Binding Energy (eV) 270.0

Report #: MSFC-02

File: PPROPMRS02	Date: 8/26/1988	Spot Size: 300 u	Flood Gun: 3.0 eV
Region 3	Disc: NASA02	# of Scans: 10	Resolution: 2

Description: POLYPROPYLENE-11, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 100 W, 90 MIN., NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-02

CONTRACTOR:	Georgia Tech Research Corporation Georgia Institute of Technology Atlanta, Georgia 30332-0420
TITLE OF REPORT:	Electron Spectroscopy for Chemical Analysis - Sample Analysis
DATE:	5 January 1989
TYPE OF REPORT:	Final
REQUISITION NUMBER:	1-7-EH-91814(1F)
GEORGIA TECH PROJECT #:	E18-654
AUTHOR:	W. B. Carter

PREPARED FOR:

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This Final Report consists of the two interem reports which were prepared during the course of the project: Reports MSFC-01 and MSFC-02. All spectra are presented in a single appendix.

REPORT MSFC-01

I. INTRODUCTION

This report describes the results of the ESCA analysis of the specimens listed in Table 1, which were supplied by MSFC.

TABLE 1

Specimens Analyzed with ESCA

Specimen	Exposure Conditions in Atomic Oxygen
KAPTON-4	Control (no exposure)
KAPTON-5	Exposed at 45°C, 60 W, 30 Min.
KAPTON-6	Exposed at 45°C, 60 W, 95 Min.
HDPE-19	Control (no exposure)
HDPE-20	Exposed at 45°C, 60 W, 30 Min.
HDPE-21	Exposed at 45°C, 60 W, 95 Min.
PVDF-4	Control (no exposure)
PVDF-5	Exposed at 45°C, 60 W, 30 Min.
PVDF-6	Exposed at 45°C, 60 W, 95 Min.

ESCA was performed on an SSL-100/206 Small Spot ESCA spectrometer. This instrument utilizes monochromatized Al Kα x-rays that are focused to a spot on the specimen. All data were taken with the use of a low voltage electron flood gun and a charge neutralization screen to minimize charging effects on the data. The charge neutralization screen is made of nickel and thus Ni photoionization peaks are seen in some of the spectra. Oxygen on this screen may produce small O 1s peaks. Nickel peaks and any possible contributions to the O 1s peak from the nickel screen have been neglected in the analyses that follow.

The voltage of the low energy electron flood gun was set by collecting C 1s spectra and adjusting the flood gun voltage to the nearest integral voltage setting that minimized the peak widths. Slight charging effects can be seen in a few of the

spectra as a small shoulder on the low binding energy side of the effected peaks.

Because the specimens are electrically insulating and charged in the x-ray beam, it was not possible to obtain an independent binding energy reference. The C 1s photoionization peak obtained from HDPE is often assigned a binding energy of about 285 eV. This peak can thus be used as an internal binding energy reference for the HDPE specimens. Such an approach is difficult to apply to Kapton and PVDF. The C 1s spectra of Kapton contain several component peaks that make the assignment of an internal binding energy reference difficult. Although PVDF contains CH_2 bonds (as does HDPE), the secondary substituent effects of F alter the C 1s binding energies.

The x-ray spot size and electron flood gun voltage used are recorded on the individual spectra as are the instrumental resolutions. Two resolutions were utilized, resolutions 4 and 2. Resolution 4 is the lowest resolution available on the instrument while resolution 2 is the second greatest available.

Two types of spectra were obtained for each specimen:

1. General Surveys

   General surveys were taken with the 1000 μm diameter spot (the largest available) at resolution 4 from a binding energy of -10 eV to 1090 eV. This 1100 eV range is the largest of which the spectrometer is capable.

2. High Resolution Spectra

   High resolution spectra were obtained of the most intense photoionization peaks for the major elements represented in the general surveys. These spectra were taken at resolution 2 using a 300 μm diameter x-ray spot. The high resolution spectra span a binding energy range of 20 eV.

Two types of data reduction were performed:

1. Semiquantitative Compositional Analysis

General surveys were analyzed semiquantitatively for the atomic composition of the specimen surfaces. This was performed with the aid of the spectrometer software which takes into account relative photoionization cross sections for the atomic levels involved and the instrument response. The effect of electron mean free path variation, which may be as large as a factor of 2, is ignored

The major photoionization peaks are labeled on the general surveys as are the major Auger peaks. The Auger peaks are indicated by an "A" following the elemental symbol.

2. Peak Fitting

The high resolution spectra were resolved into the fewest number of Gaussian components required for a reasonable fit. C 1s components were of FWHM 1.35 eV, and the O 1s components were of FWHM 1.84 eV. The widths of the N 1s and F 1s components were about 1.8 and 2.1 eV, respectively.

II. DATA ANALYSIS

A. Compositional Analysis.

The spectra collected are presented in the appendix. Table 2 lists the composition of the surface of each specimen as determined semiquantitatively from its general survey. The atomic percents are given to the nearest percent and thus may not sum to 100%.

B. Analysis of Specimens: KAPTON-4, KAPTON-5, and KAPTON-6.

The general survey from the control specimen (KAPTON-4) indicates that the specimen surface is composed primarily of C, O, and N (the constituents of KAPTON H). The C 1s spectrum of this specimen resolves into three components: 1. an aromatic

component at a binding energy of about 281 eV due to the C atoms bonded to only C and H, 2. an unresolved aromatic component at about 282.2 eV, which is due to C atoms singly bonded to O or N, and 3. a component due to carbonyl type bonds (C=O). The C 1s spectrum is similar to those for untreated KAPTON H in the published literature.[1]

There appears to be a single component to the N 1s spectrum of the unexposed specimen and two components of approximately equal magnitude to its O 1s spectrum. The O 1s component at the greatest binding energy corresponds to the ether bonded O (C-O-C) while the component at the least binding energy is due to carbonyl bonded O (C=O). O 1s spectra in the literature display a carbonyl component that is about twice the size of the ether component in contradiction to what is seen here (see footnote).

TABLE 2

Surface Composition of Polymer Specimens

Specimen	Atomic Concentration (%)			
	C	N	O	F
KAPTON-4	78	6	16	-
KAPTON-5	70	6	24	-
KAPTON-6	67	7	25	-
HDPE-19	97	-	3	-
HDPE-20	83	-	17	-
HDPE-21*	84	-	15	-
PVDF-4	56	-	1-2	42
PVDF-5	53	-	5	42
PVDF-6	54	-	5	41

* HDPE-21 had about 1% Ca on its surface.

[1] M. Kogoma and G. Turban, Plasma Chemistry and Plasma Processing, 6 (4), (1986) 349.

Both general survey spectra of the plasma exposed specimens (KAPTON-5 and KAPTON-6) indicate an increased O concentration relative to the unexposed specimen. The C 1s spectra of these specimens consist of several components: 1. two components at low binding energies, which are due to the C-C-C, and the C-O and C-N bonds in KAPTON H, and 2. possibly three components at greater binding energies, which are due to C atoms in increased oxidation states. The proportion of C atoms in the greater oxidation states, i.e., greater than those of C-C-C, C-O, and C-N, is larger after plasma exposure that before. These three components represent about 89 percent of the C 1s signal prior to plasma exposure and about 64 percent after plasma exposure.

The N 1s spectra taken from the plasma exposed KAPTON H specimens appear to be comprised of two components. The separation between the resolved peaks is about 1.1 eV. The component at the greater binding energy may be due N-O bonds.

The O 1s spectra from the plasma exposed specimens resolve into two components of approximately equal magnitude. The existence of two resolvable components after exposure to an RF oxygen plasma is consistent with the literature as is the fact that their relative magnitudes are unchanged by the plasma (see footnote).

C. Analysis of Specimens: HDPE-19, HDPE-20, and HDPE-21.

The general survey from the control specimen (HDPE-19) indicates that the specimen surface is primarily composed of C and O. As there should be no O in the pure polymer, the O may be due to a slight oxidation of the surface and/or contamination. The solvent used to clean the specimen may have been the source of the O observed. The C 1s spectrum of this specimen contains two (2) components: 1. a major peak due to CH_2 bonds in HDPE that appears at a binding energy of about 280 eV (shifted due to charging) and 2. a small peak shifted upwards in binding energy from the major peak by about 4.2 eV that is probably due to oxidation of C.

Both general survey spectra of the plasma exposed specimens (HDPE-20 and HDPE-21) indicate an increased O concentration relative to the unexposed specimen. Both the relative and absolute increase in surface O concentration after plasma exposure are greater for the HDPE than for either KAPTON H or PVDF (see TABLE 2).

The C 1s spectra of the plasma exposed specimens consist of several components: 1. a major component due to $\underline{C}H_2$ bonds, and 2. several components at larger binding energies that correspond to C in various states of oxidation. The differences between the spectra from these two specimens cannot be considered significant since only one spectrum of each type was taken from each specimen.

D. Analysis of Specimens: PVDF-4, PVDF-5, and PVDF-6.

The general survey from the control specimen (PVDF-4) indicates that the specimen surface is composed of primarily F and C. There is a minor amount of O, which is probably due to contamination.

The C 1s spectrum of the unexposed specimen consists of two resolvable components separated by about 4.5 eV. That at the lowest binding energy is due to $\underline{C}H_2$ and that at the highest binding energy is due to $\underline{C}F_2$. It appears from the FWHM of these components that each is probably composed of several unresolved components due to various nonideal structures in the polymer, eg., $\underline{C}HF$.

The F 1s spectrum of the unexposed specimen is fit well with a single Gaussian peak. The O 1s spectrum resolves into two components separated by 1.0 eV.

Both general survey spectra of the plasma exposed specimens (PVDF-5 and PVDF-6) indicate an increased O concentration relative to the control specimen.

The C 1s and F 1s spectra of the plasma exposed specimens are similar to those of the unexposed specimen. The separation between these photoionization peaks also is unchanged by plasma

exposure. The O 1s spectra of the plasma exposed specimens resolve into two components of similar magnitude and separated by about 1.4 eV.

From this data, it is not possible to determine whether the oxidation state of C is changed by plasma exposure.

III. SUMMARY

Plasma treatment appears to oxidize all of the polymers examined. Evidence for this are the increases in O on the specimen surfaces after plasma exposure and the increases in the relative amounts of C in various oxidized states following exposure.

Although the relative oxygen increase of the PVDF was greater than that of the KAPTON H specimen, the absolute increase for the PVDF was the smallest observed (see TABLE 2 and recall that KAPTON H contains structural O). The PVDF specimens displayed the least O both prior to plasma exposure and after plasma exposure. Absolute amounts of O due to plasma exposure increase in the order: PVDF < KAPTON < HDPE.

I. INTRODUCTION

This report describes the results of the ESCA analysis of the specimens listed in Table 1, which were supplied by MSFC. ESCA data collection and reduction were performed as described in Report # MSFC-01 dated 23 August 1988.

TABLE 1

Specimens Analyzed with ESCA

Specimen	Exposure Conditions in Atomic Oxygen
MYLAR CONTROL	Control (no exposure)
MYLAR-11	Exposed at 45 ^{0}C, 60 W, 75 Min.
POLYPROPYLENE CONTROL	Control (no exposure)
POLYPROPYLENE-11	Exposed at 45 ^{0}C, 100 W, 90 Min.

II. DATA ANALYSIS

A. Compositional Analysis.

The spectra collected are presented in the appendix. Table 2 lists the composition of the surface of each specimen, with the exception of specimen Mylar-11, as determined semiquantitatively from its general survey. The surface of Mylar-11 has a substantial amount of Sb on it. Since the Sb 3d5/2 photoionization peak overlaps the O 1s photoionization peak, quantification was not possible. The Mylar control specimen may have a small amount of Sb on it (see below), which has been ignored in the quantification. The atomic percents are given to the nearest percent and thus may not sum to 100%.

B. Analysis of Specimens: MYLAR AND MYLAR-11.

The general survey from the control specimen (MYLAR) indicates that the specimen surface is composed primarily of C and O, with a trace of F and possibly Sb contamination. The C 1s

spectrum of this specimen resolves into five components.

TABLE 2

Surface Composition of Polymer Specimens

Specimen	Atomic Concentration (%) C	O	F	Al	Na
MYLAR	75	24	0.5	-	-
MYLAR-11 *					
POLYPROPYLENE	97	3	-	-	-
POLYPROPYLENE-11	80	14	2.5	3	0.5

* The surface of MYLAR-11 consists primarily O and C, with smaller amounts of Sb, Ca, Na, N, and P.

The O 1s spectrum of the unexposed specimen resolves into two components.

The general survey spectra of the plasma exposed specimen (MYLAR-11) indicates an increased O to C ratio relative to the unexposed specimen. The C 1s spectrum of this specimen differs from that of the unexposed specimen. Assuming that the C 1s peak that appears at 282.1 eV on the control specimen is due to C in the same state as that which produces the peak at 273.9 eV on the exposed specimen, the average C 1s binding energy is greater on the exposed specimen than on the control. The lack of an absolute binding energy reference (see Report # MSFC-01) prevents a more conclusive analysis at this time.

The O 1s spectrum of the plasma exposed specimen overlaps the Sb 3d5/2 peak. Peak fitting was not attempted. The Sb 3d3/2 peak can be seen at a binding energy of about 529.5 eV.

C. Analysis of Specimens: POLYPROPYLENE and POLYPROPYLENE-11.

The general survey of the control specimen (POLYPROPYLENE) indicates that the specimen surface is composed primarily of C and O. As there should be no O in the pure polymer, the O may be due to a slight oxidation of the surface and/or contamination.

The solvent used to clean the specimen may have been the source of the O observed.

The C1s spectrum of this specimen resolves into two primary peaks due to the different bonding states of C in the polymer and a small peak at a greater binding energy. This small component may be the result of surface oxidation.

The O 1s spectrum of the control specimen resolves into two components.

The general survey spectrum of the plasma exposed specimen (POLYPROPYLENE-11) indicates an increased O to C ratio relative to the unexposed specimen. Several impurities also are present on the plasma exposed specimen, which are not seen on the control.

The C 1s spectrum of the plasma exposed specimen is broader than that from the unexposed specimen and resolves into three primary components and one or more components at greater binding energies. This structure is consistent with oxidation of the polymer.

The O 1s peak of the plasma exposed specimen resolves into two components.

III. SUMMARY

Plasma treatment appears to oxidize both of the polymers examined. Evidence for this are the increases in O on the specimen surfaces after plasma exposure and the increases in the relative amounts of C in various oxidized states following exposure.

It is not possible to quantify the relative extent of the oxidation observed because of the difficulty imposed by the presence of Sb on MYLAR-11 (see above). In any case, this would most likely be a meaningless exercise since the specimens were exposed in different systems under different conditions.

The surfaces of both plasma exposed specimens contained impurities. This may be indicative of contamination in the plasma reactors.

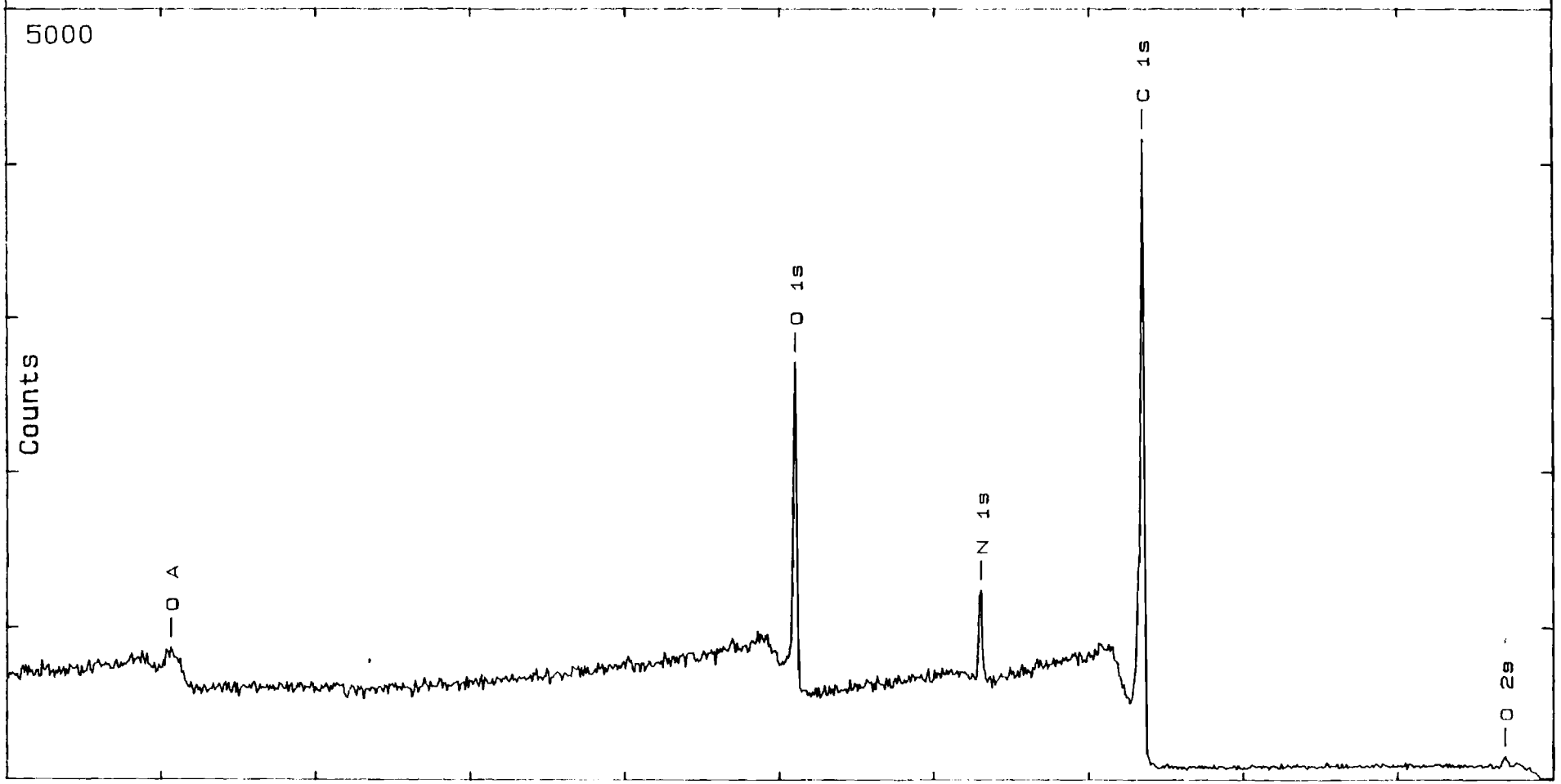
APPENDIX

ESCA SPECTRA

File: KPTNMRS12	Date: 8/16/1988	Spot Size: 1000 u	Flood Gun: 1.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4

Description: KAPTON-4, CONTROL (NO EXPOSURE)
CHARGE NEUTRALIZATION SCREEN

Operator: WBC



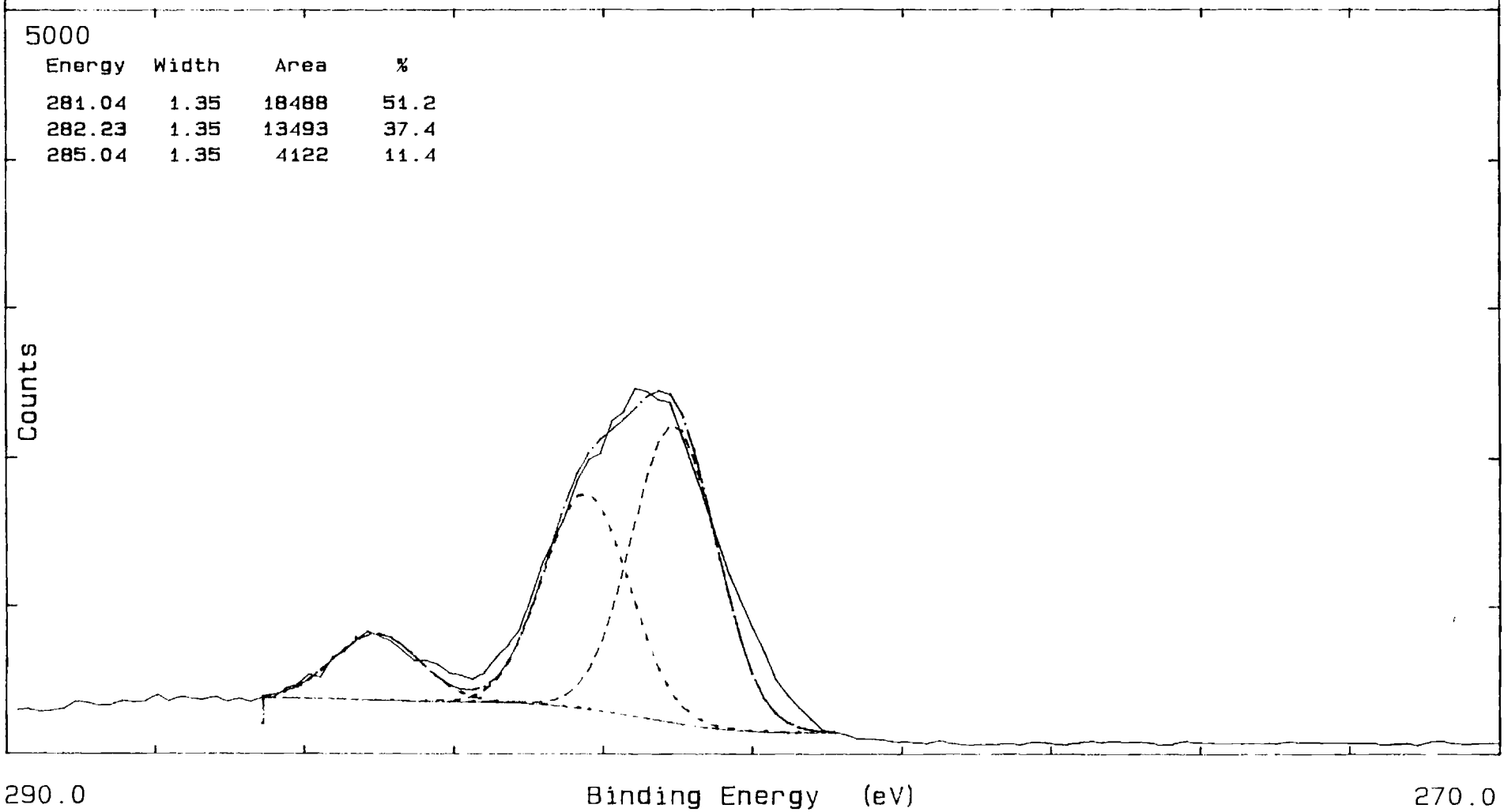
1090.0 Binding Energy (eV) -10.0

Report #: MSFC-01

File: KPTNMRS12	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 1.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: KAPTON-4 (CONTROL), C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

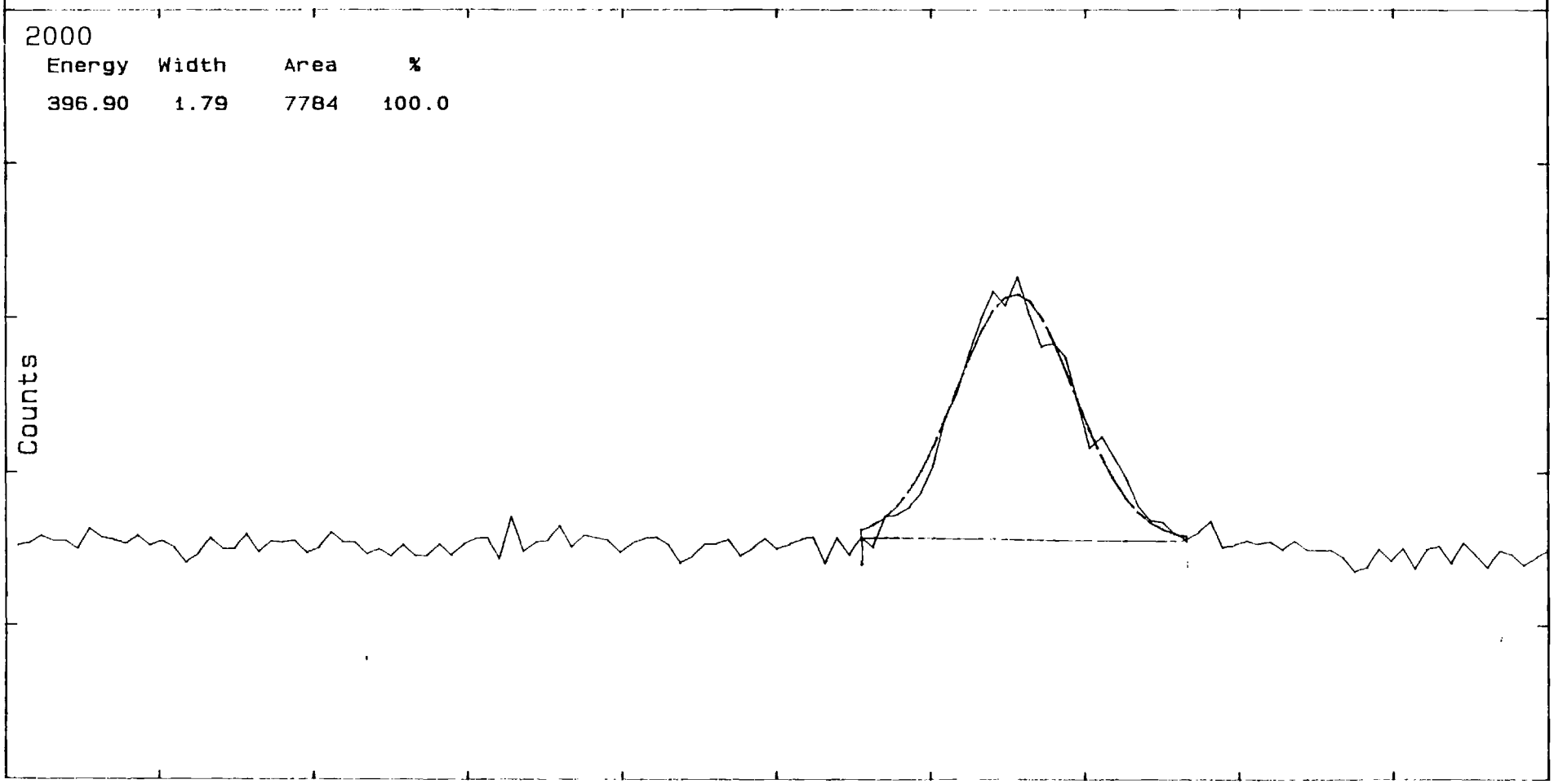


Report #: MSFC-01

File: KPTNMRS12	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 1.0 eV
Region 3	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: KAPTON-4 (CONTROL), N 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

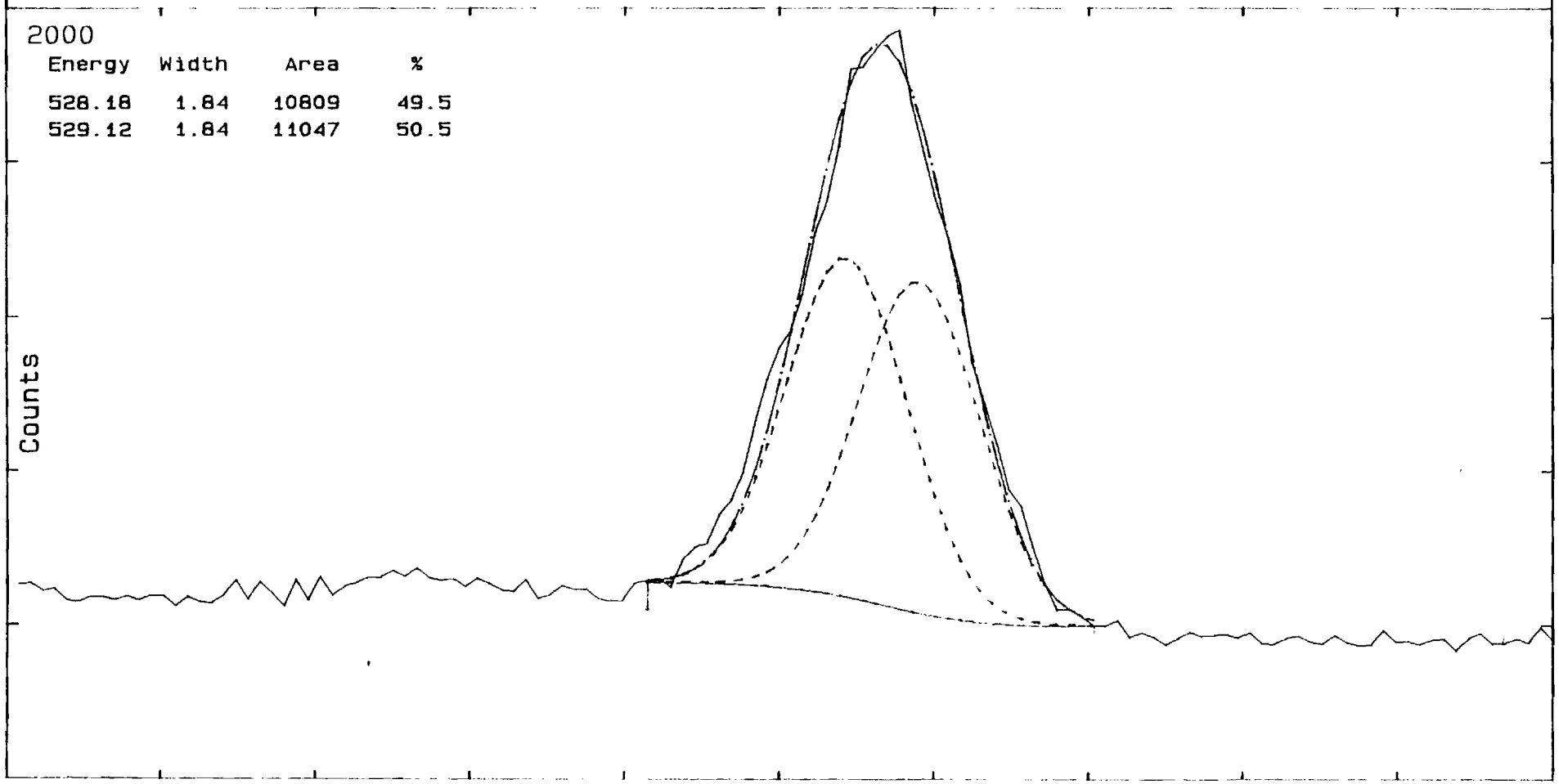


Report #: MSFC-01

File: KPTNMRS12	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 1.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: KAPTON-4 (CONTROL), O 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

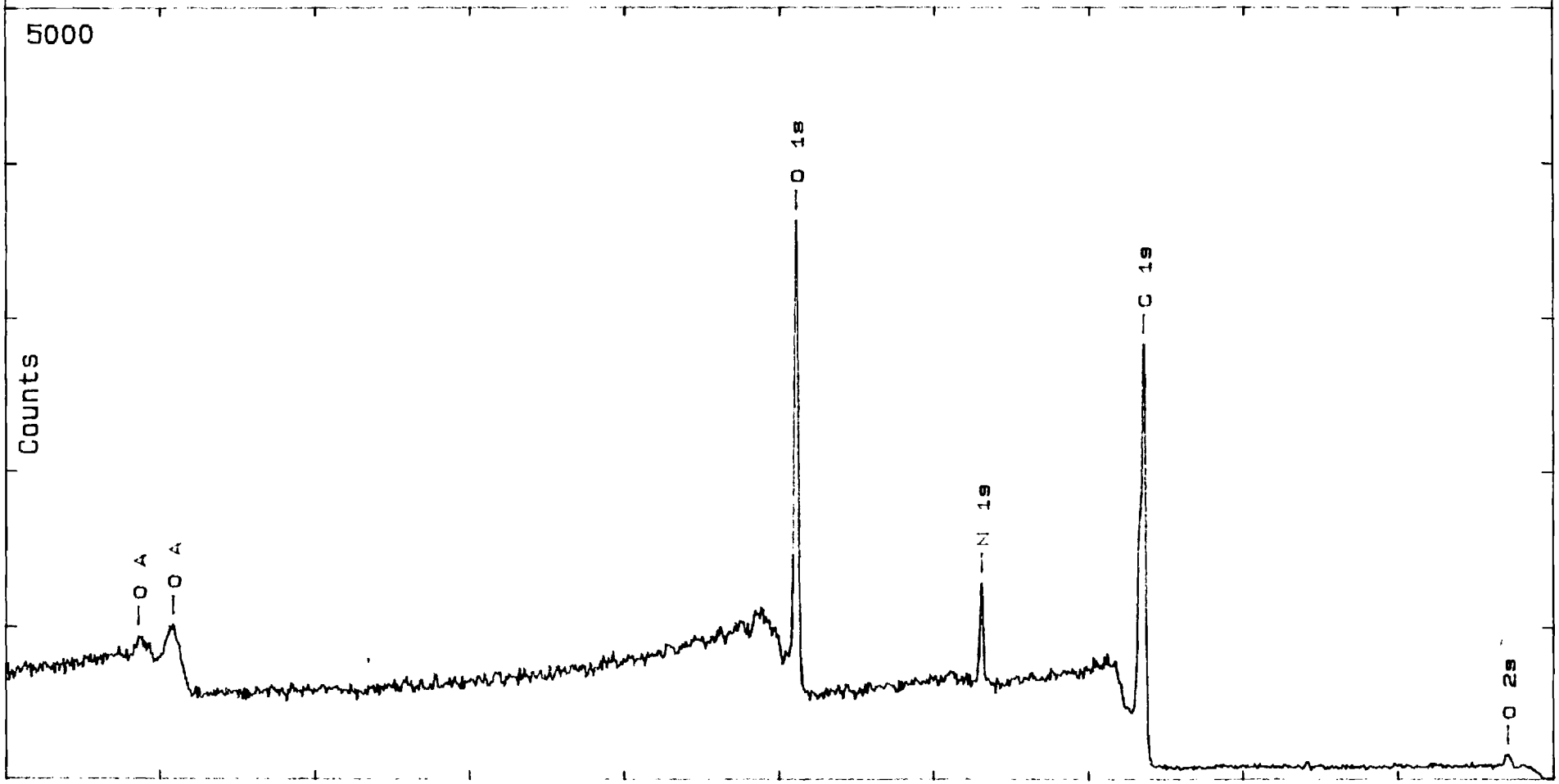


Report #: MSFC-01

File: KPTNMRS13	Date: 8/16/1988	Spot Size: 1000 u	Flood Gun: 2.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4

Description: KAPTON-5, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN. CHARGE NEUTRALIZATION SCREEN

Operator: WBC

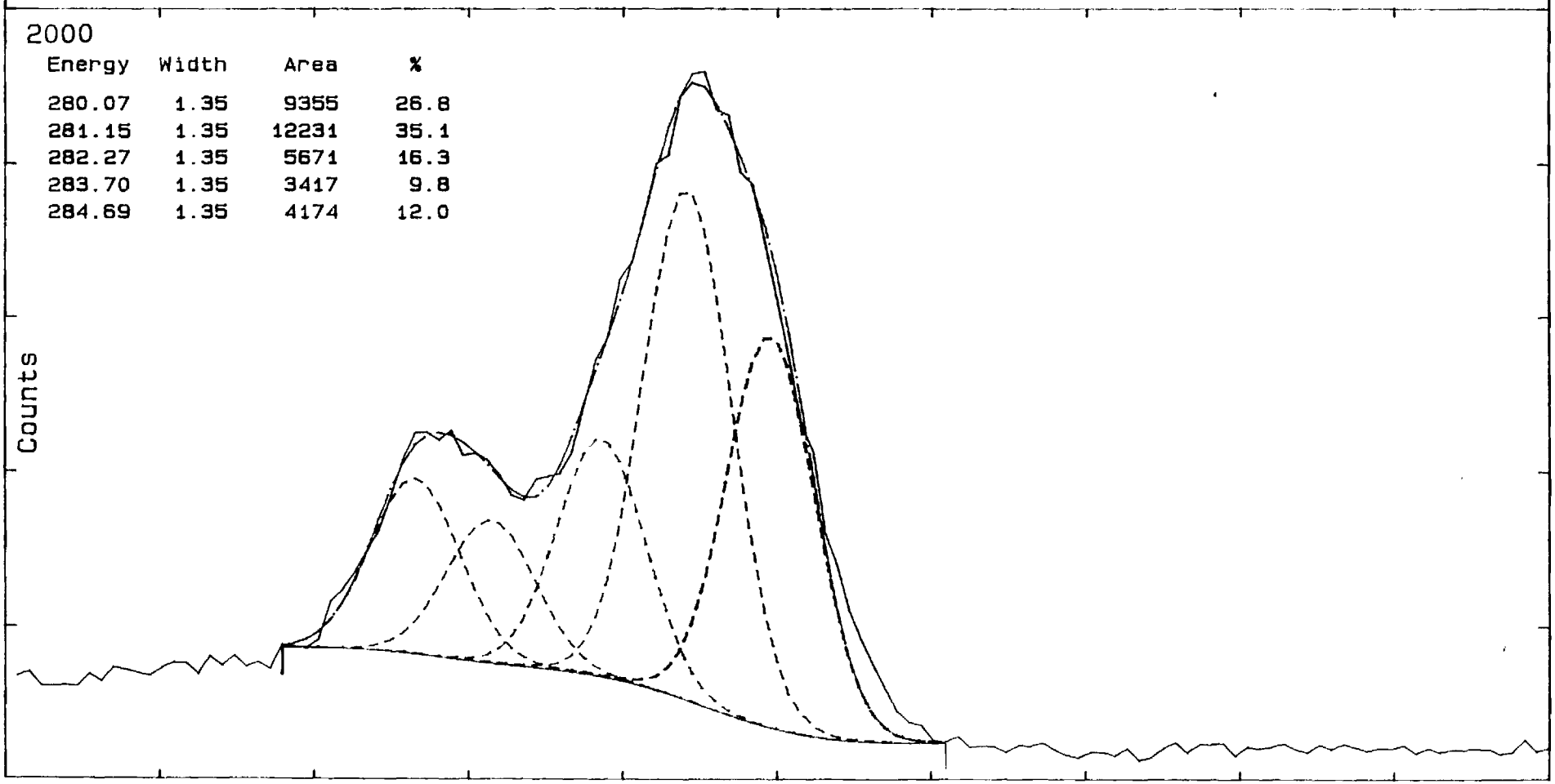


Report #: MSFC-01

File: KPTNMRS13	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: KAPTON-5, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

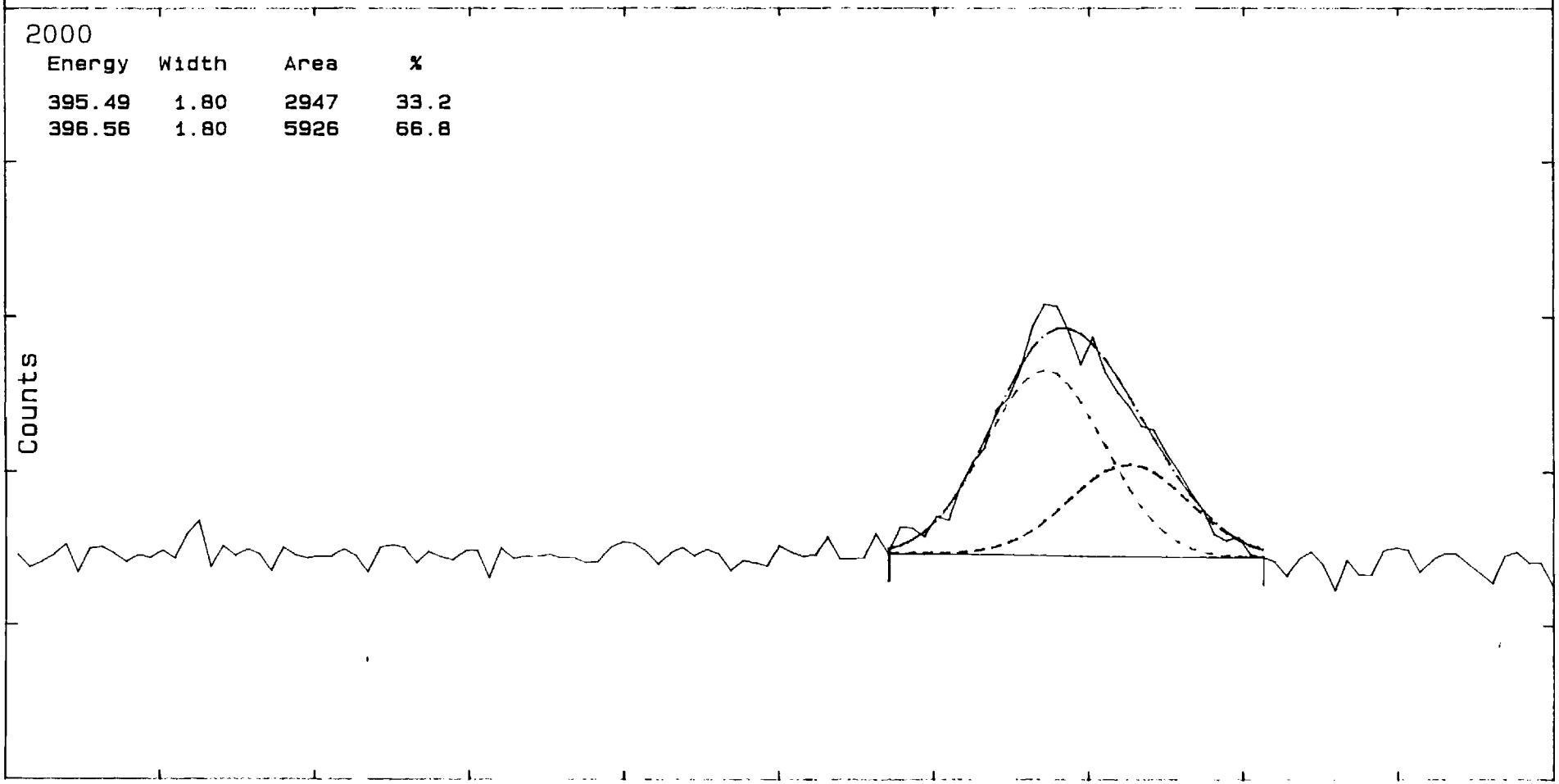


Report #: MSFC-01

File: KPTNMRS13	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 3	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: KAPTON-5, N 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

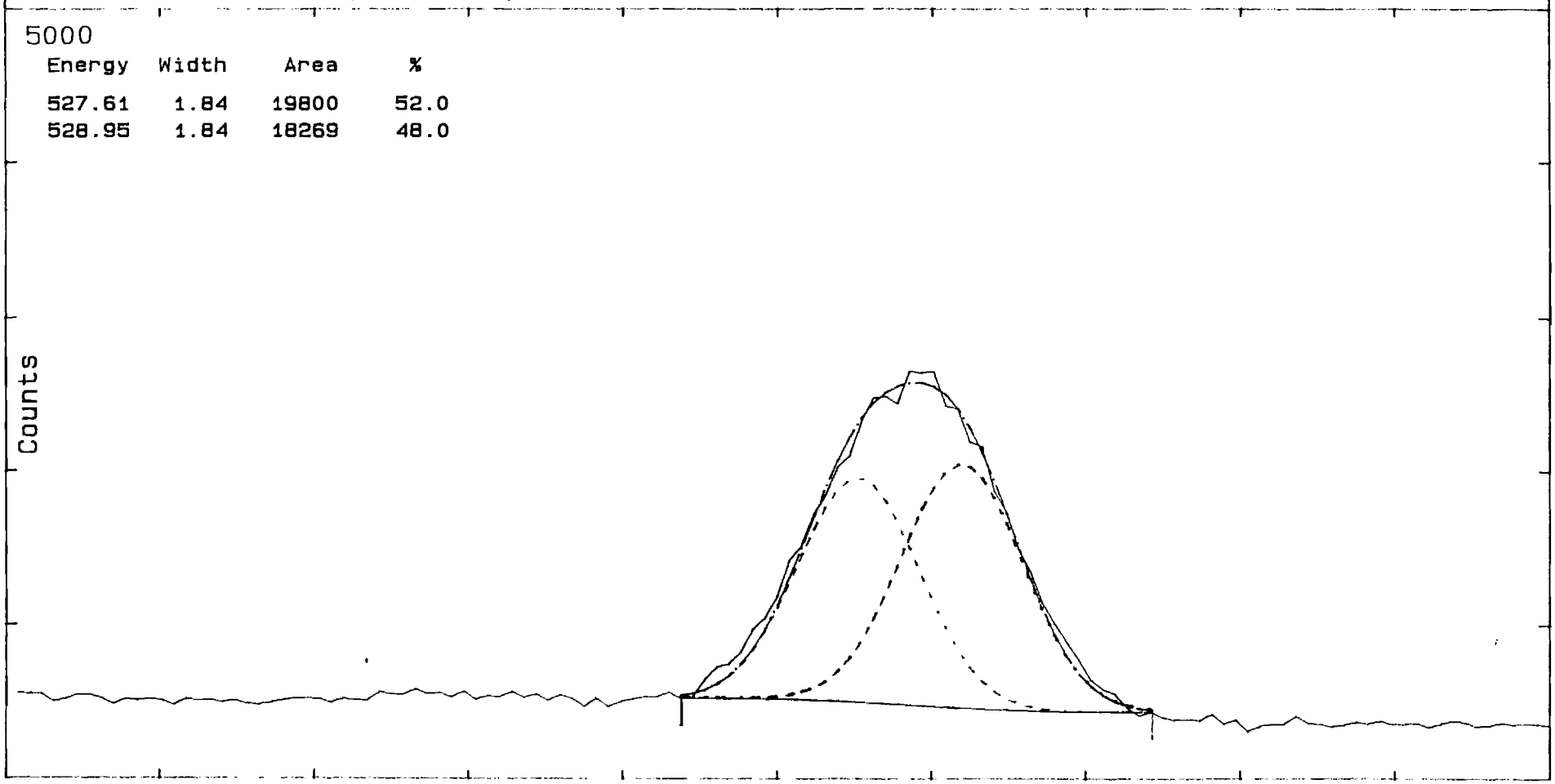


Report #: MSFC-01

File: KPTNMRS13	Date: 8/16/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2

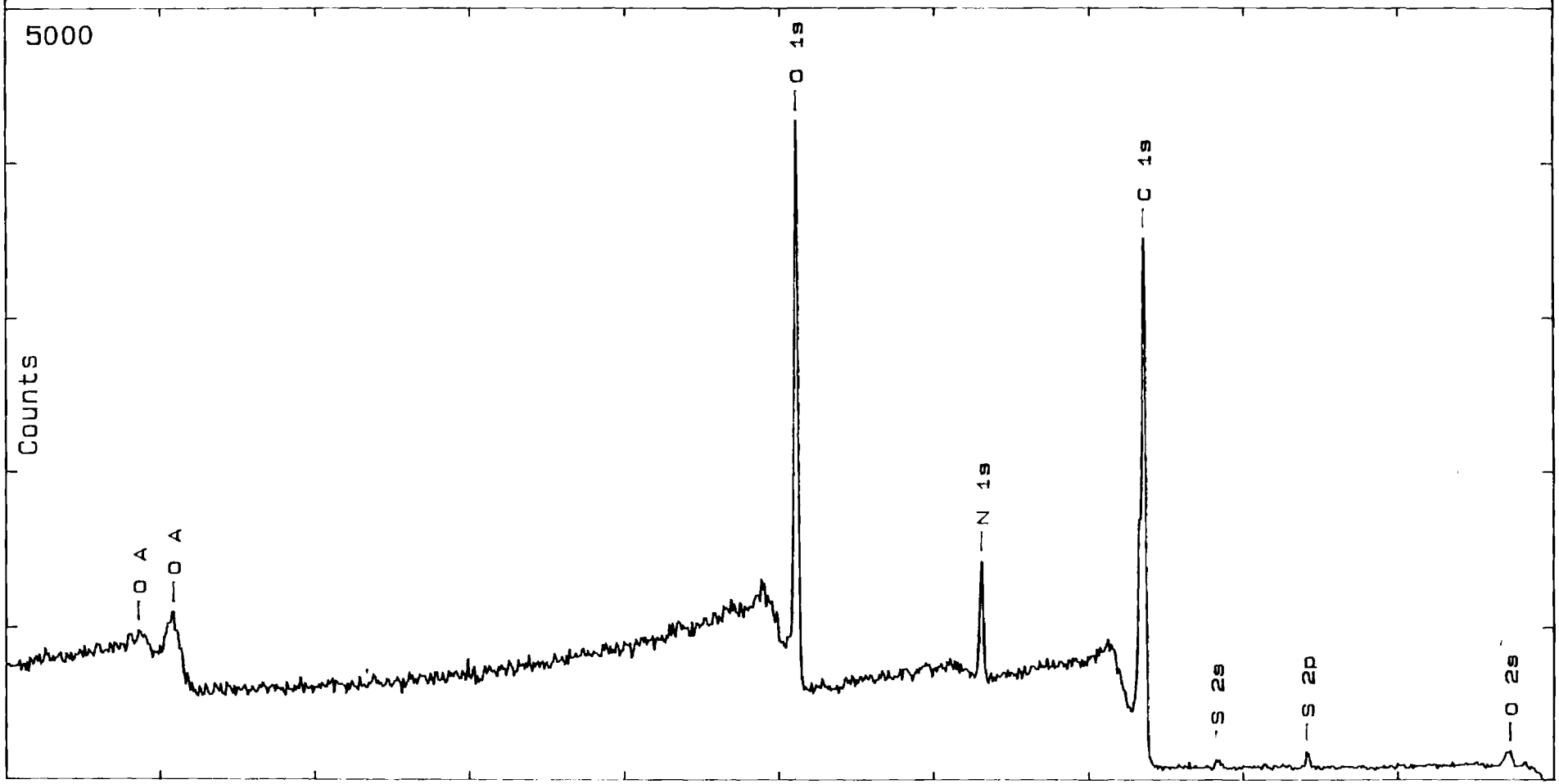
Description: KAPTON-5, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

File: KPTNMRS14	Date: 8/17/1988	Spot Size: 1000 u	Flood Gun: 2.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4
Description: KAPTON-6, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN. CHARGE NEUTRALIZATION SCREEN			Operator: WBC



Report #: MSFC-01

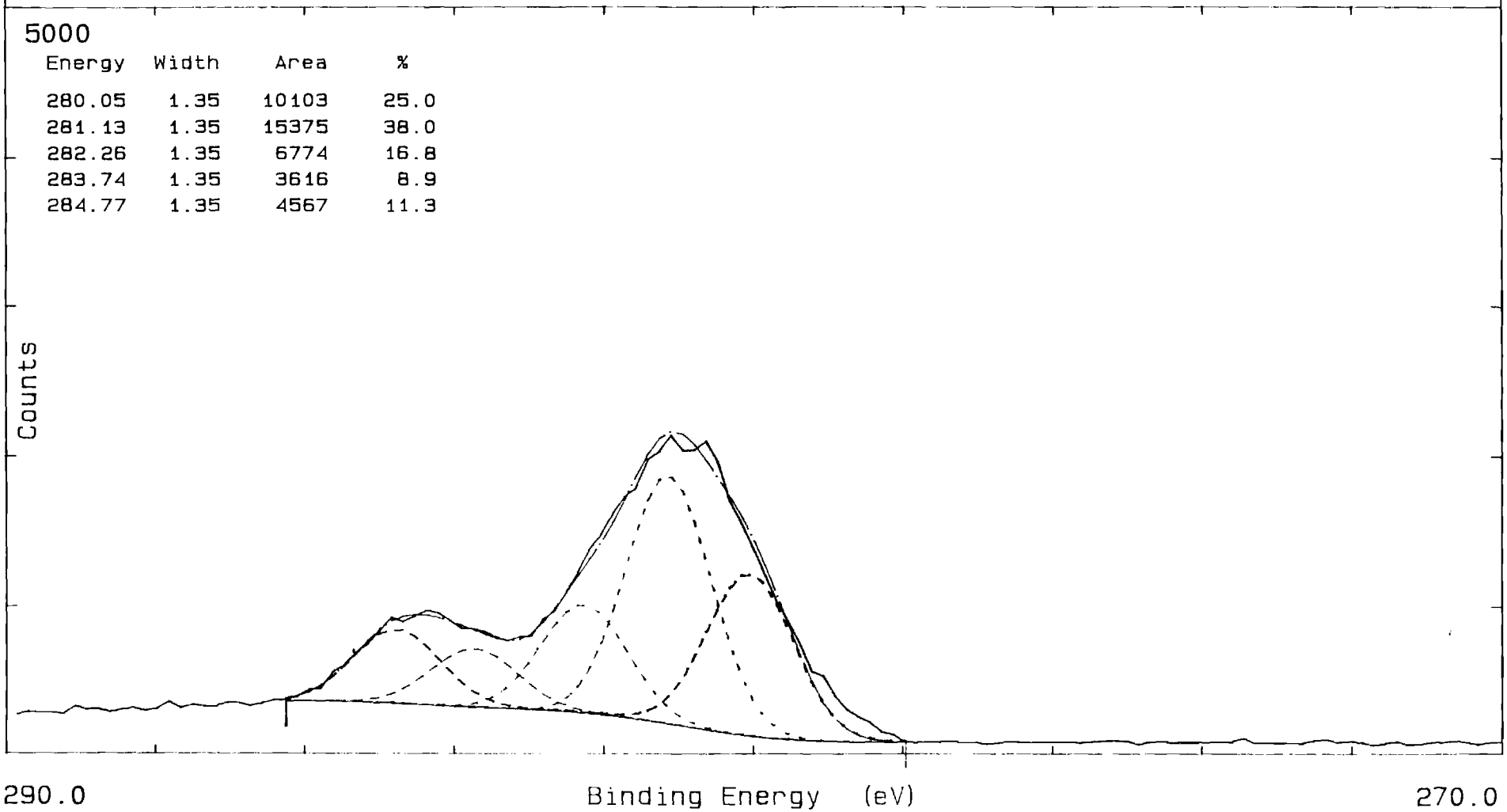
File: KPTNMRS14	Date: 8/17/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: KAPTON-6, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

5000

Energy	Width	Area	%
280.05	1.35	10103	25.0
281.13	1.35	15375	38.0
282.26	1.35	6774	16.8
283.74	1.35	3616	8.9
284.77	1.35	4567	11.3

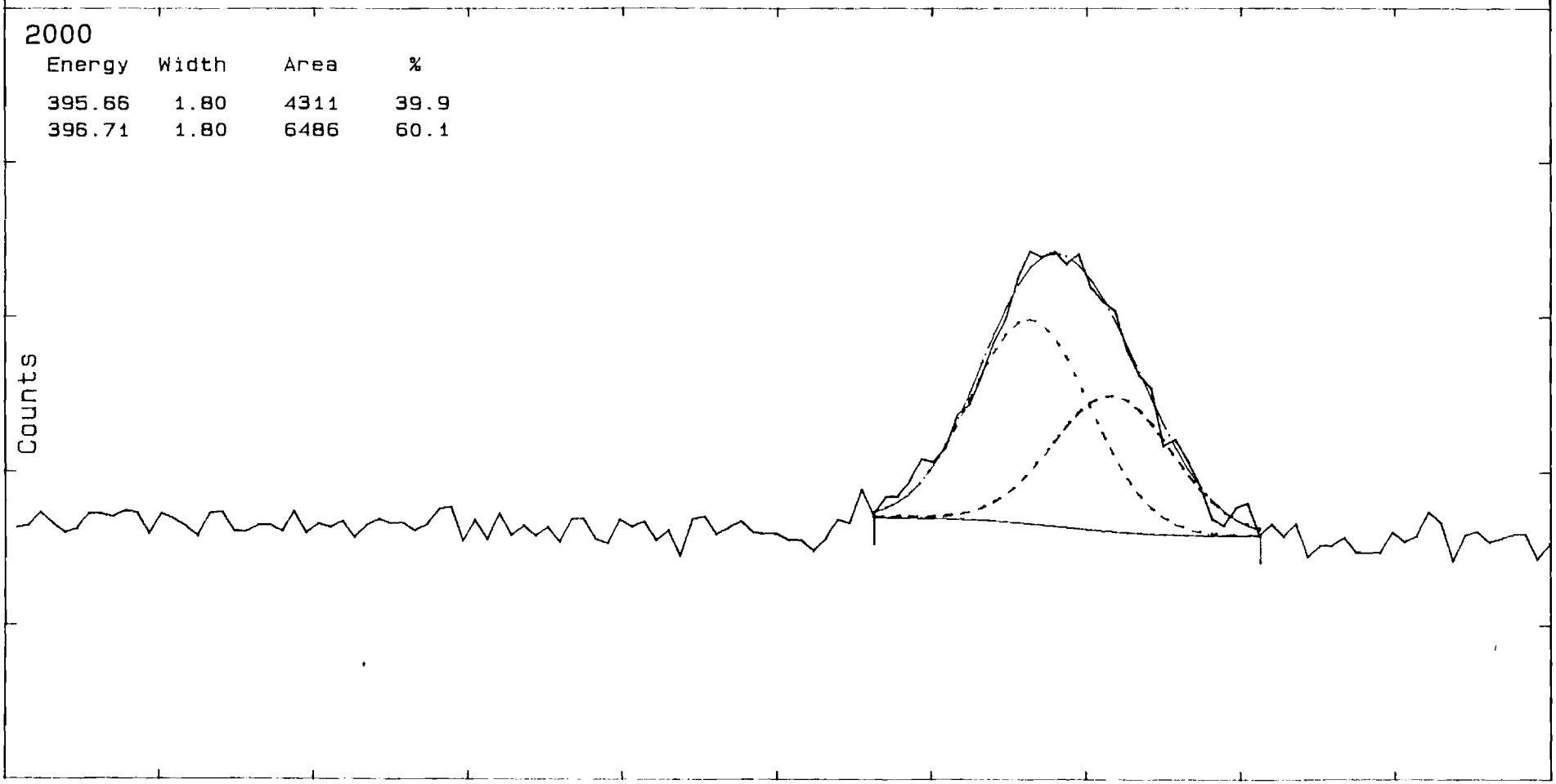


Report #: MSFC-01

File: KPTNMRS14	Date: 8/17/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 3	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: KAPTON-6, N 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

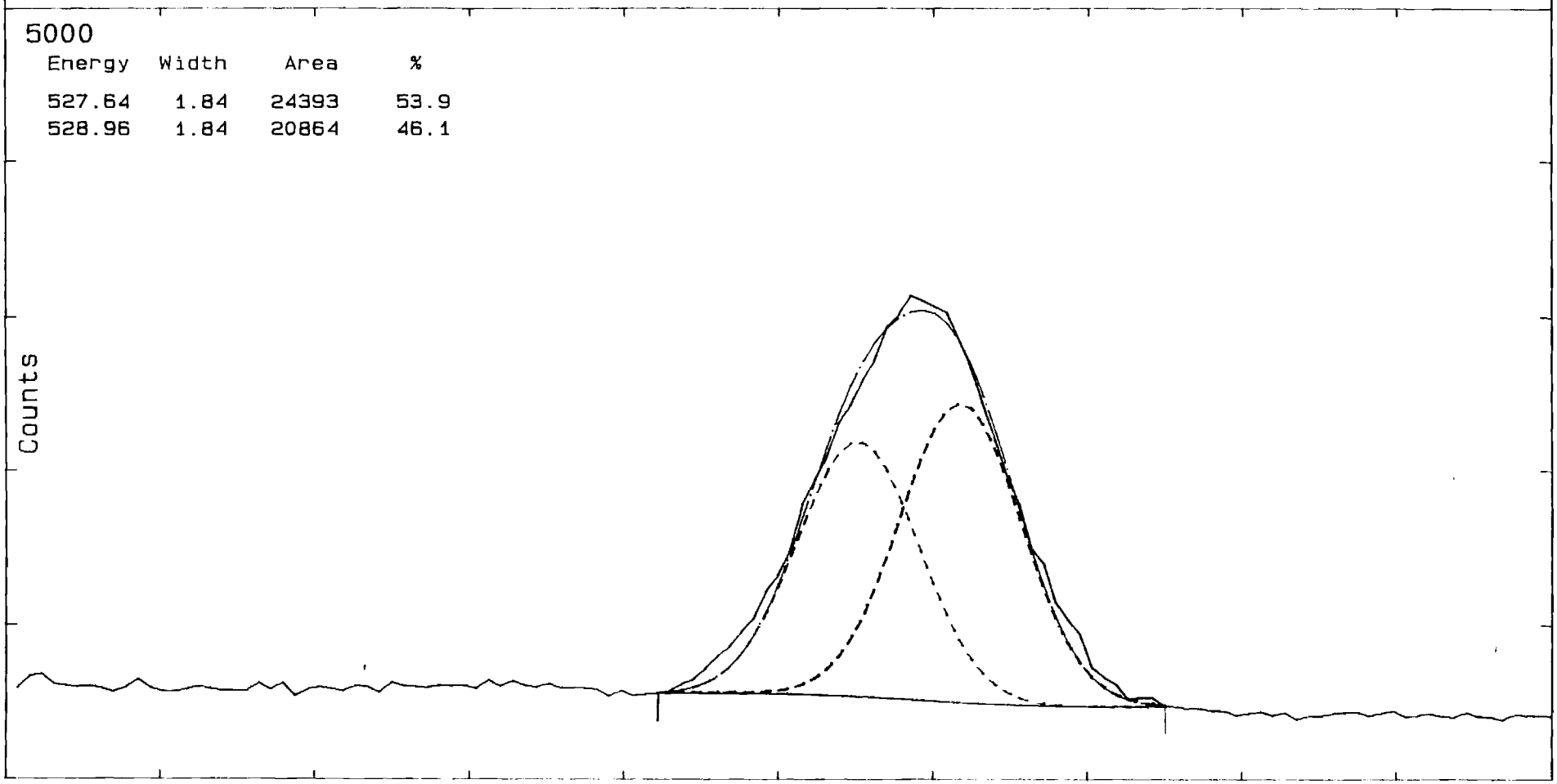


Report #: MSFC-01

File: KPTNMRS14	Date: 8/17/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: KAPTON-6, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

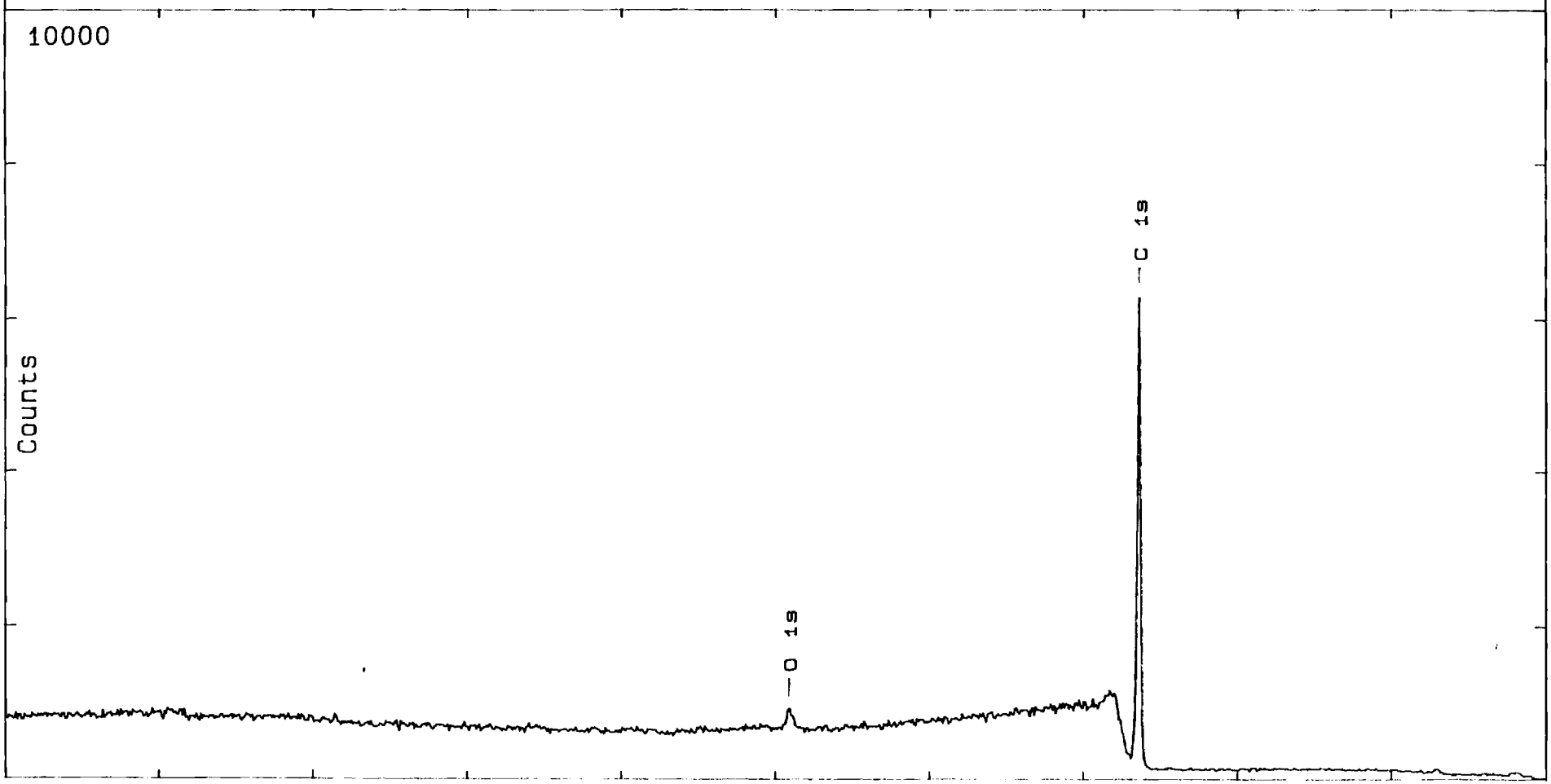


Report #: MSFC-01

File: HDPE10	Date: 8/15/1988	Spot Size: 1000 u	Flood Gun: 2.0 eV
	Disc: NASA01	# of Scans: 1	Resolution: 4

Description: HDPE-19, CONTROL (NO EXPOSURE)
CHARGE NEUTRALIZATION SCREEN

Operator: WBC



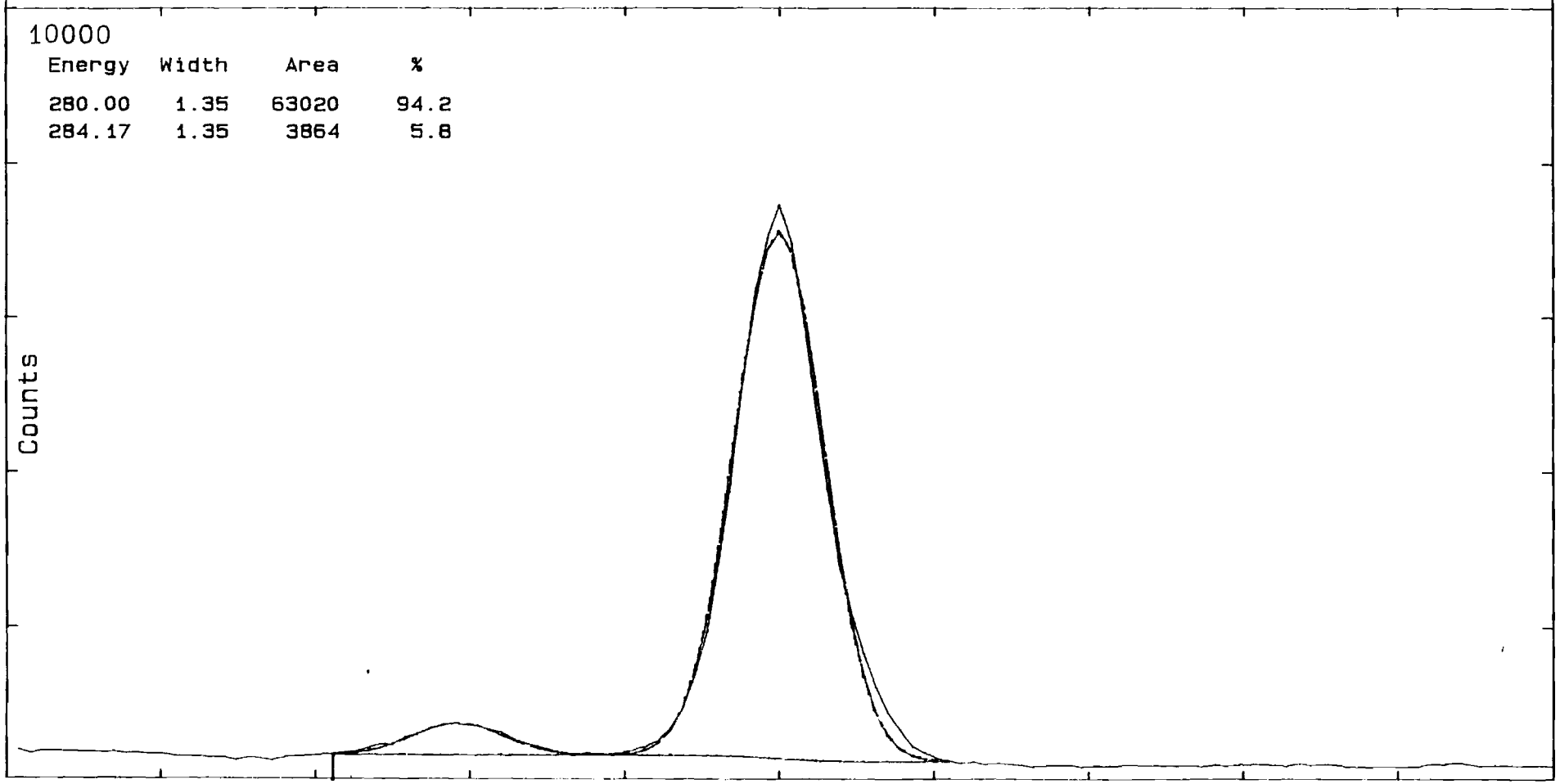
1090.0 Binding Energy (eV) -10.0

Report #: MSFC-01

File: HDPEMRS11	Date: 8/15/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 1	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: HDPE-19 (CONTROL), C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

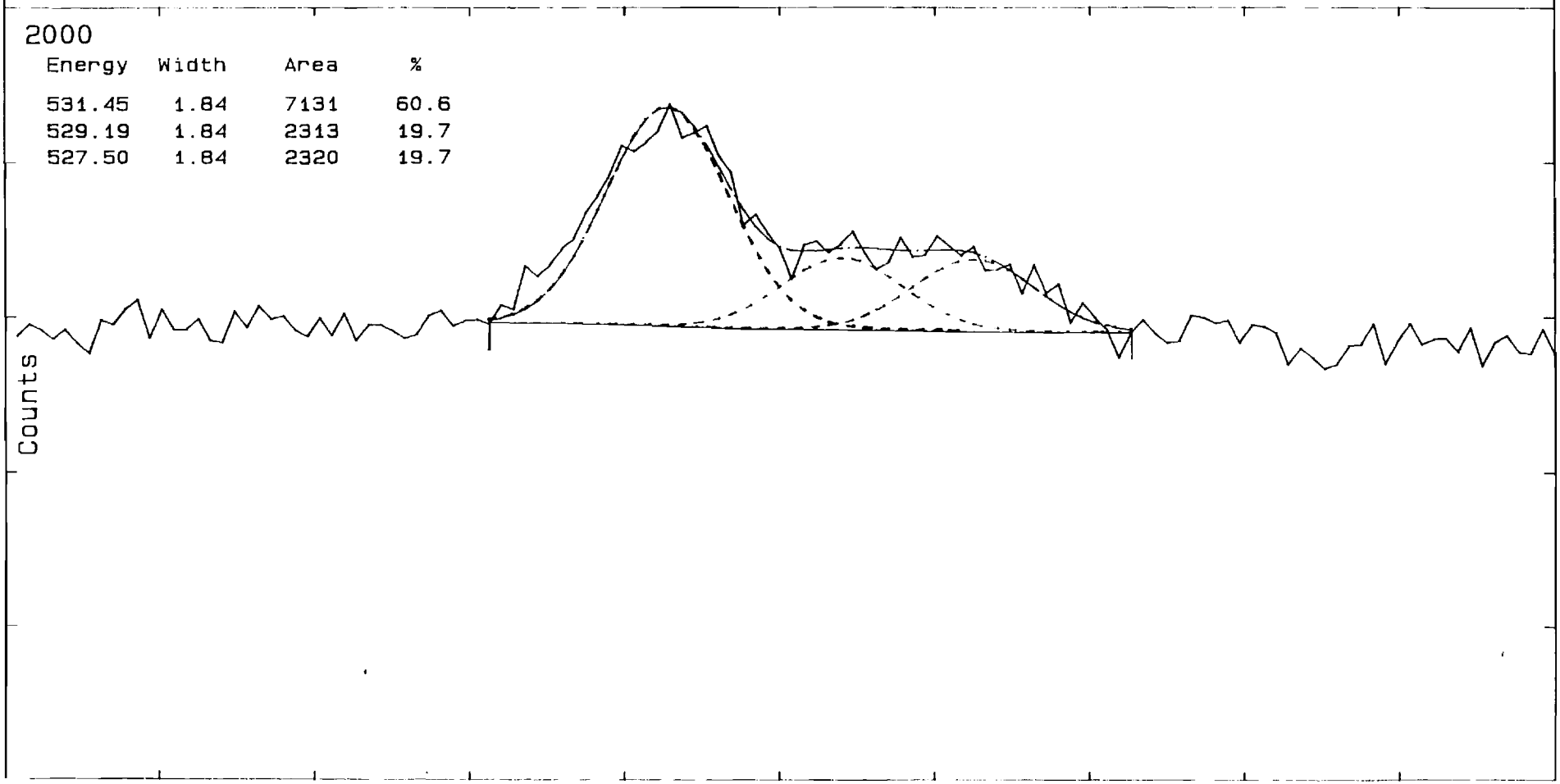


Report #: MSFC-01

File: HDPEMRS11	Date: 8/15/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 2	Disc: NASA01	# of Scans: 30	Resolution: 2

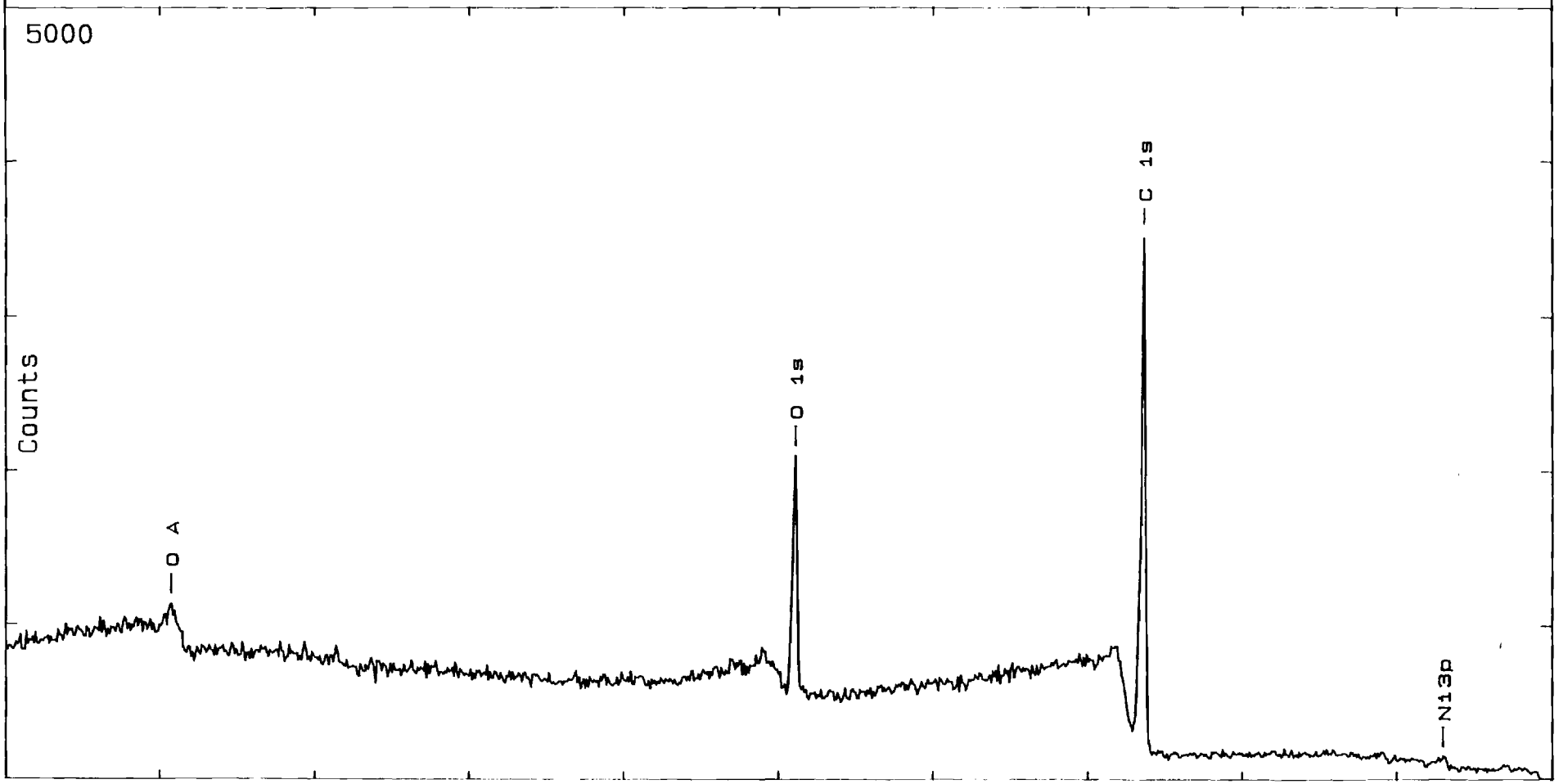
Description: HDPE-19 (CONTROL), O 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

File: HDPE12	Date: 8/15/1988	Spot Size: 1000 u	Flood Gun: 2.0 eV
	Disc: NASA01	# of Scans: 1	Resolution: 4
Description: HDPE-20, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN. CHARGE NEUTRALIZATION SCREEN			Operator: WBC



Report #: MSFC-01

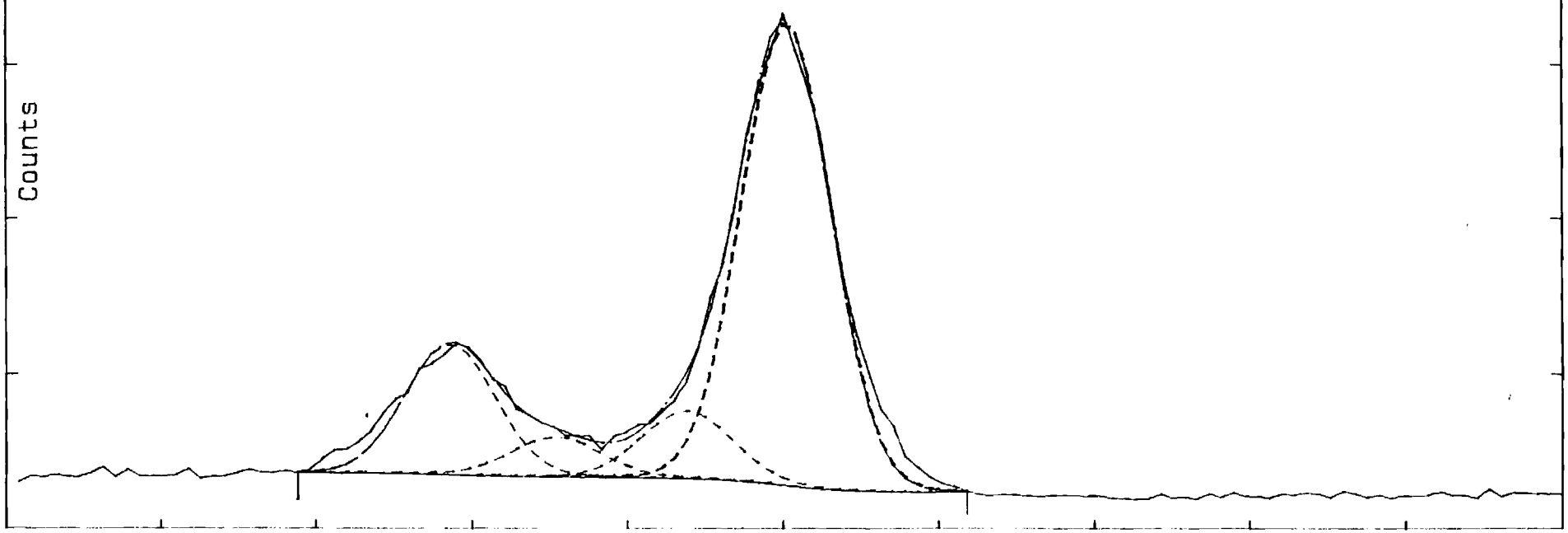
File: HDPEMRS13	Date: 8/15/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 1	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: HDPE-20, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN.; CHARGE NEUTRALIZATION SCREEN

Operator: WBC

5000

Energy	Width	Area	%
279.96	1.35	27340	66.2
281.25	1.35	3970	9.6
282.94	1.35	2329	5.6
284.29	1.35	7683	18.6



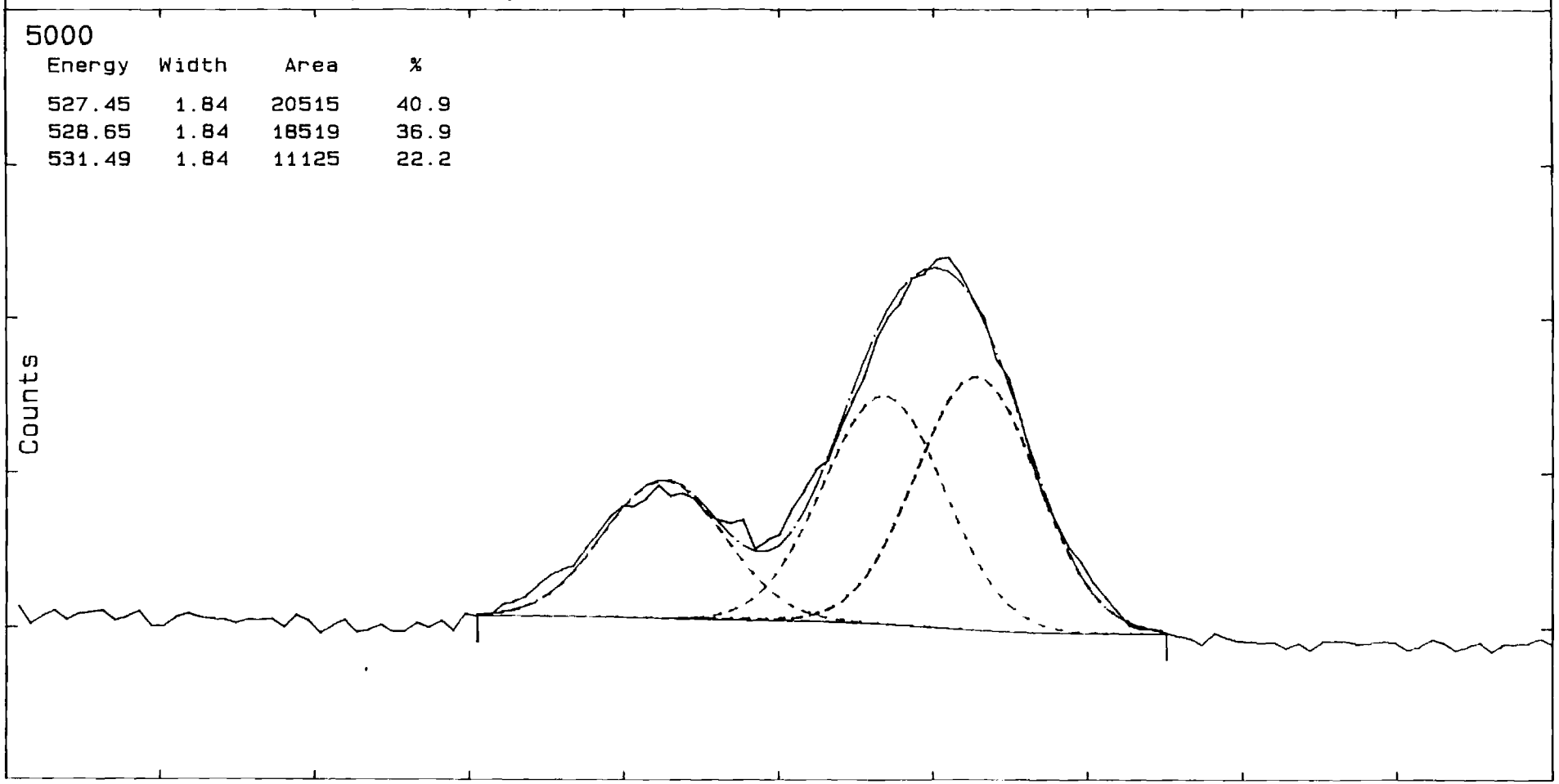
290.0 Binding Energy (eV) 270.0

Report #: MSFC-01

File: HDPEMRS13	Date: 8/15/1988	Spot Size: 300 u	Flood Gun: 2.0 eV
Region 2	Disc: NASA01	# of Scans: 30	Resolution: 2

Description: HDPE-20, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN.; CHARGE NEUTRALIZATION SCREEN

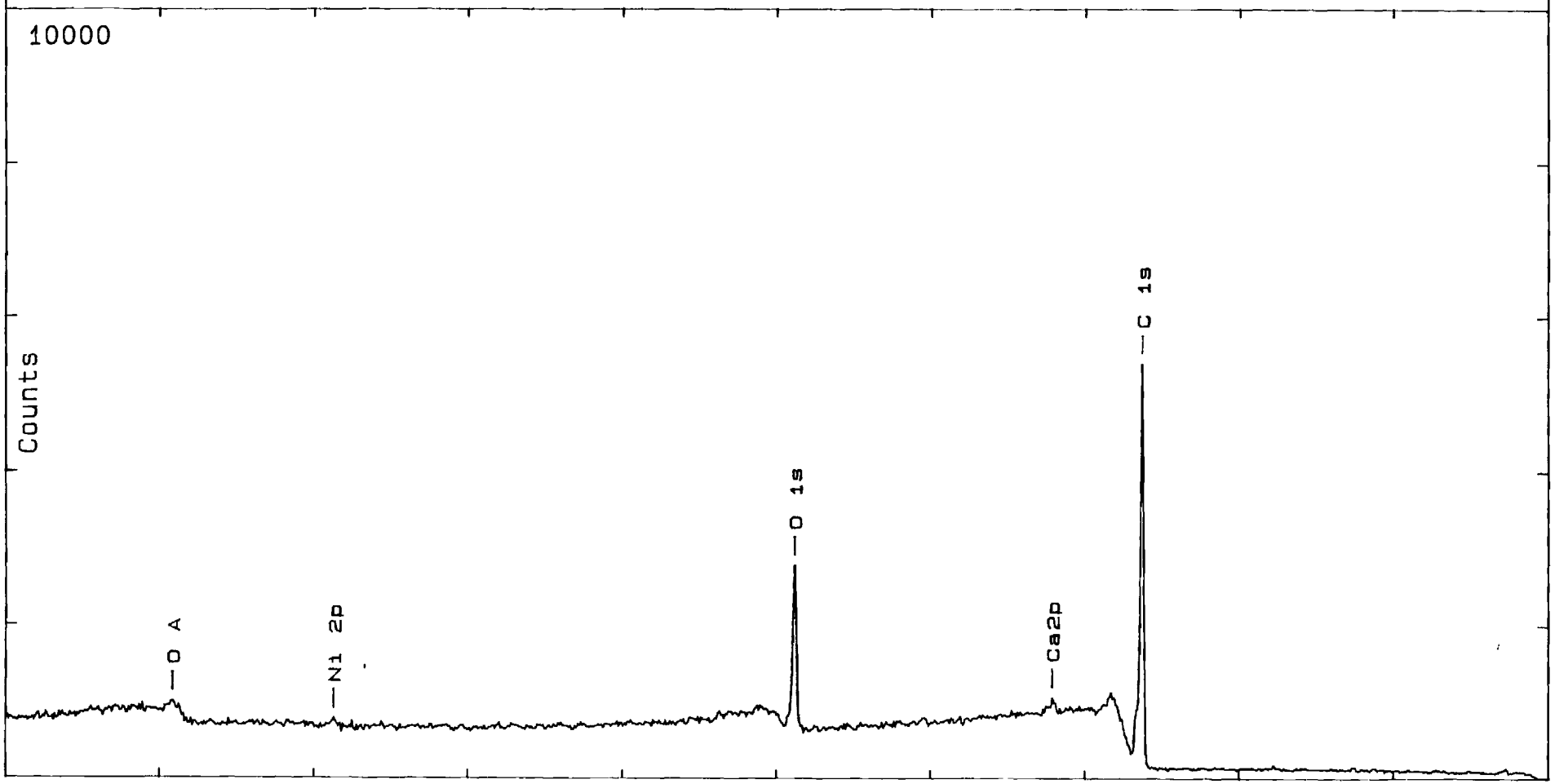
Operator: WBC



Report #: MSFC-01

File: HDPE14	Date: 8/15/1988	Spot Size: 1000 u	Flood Gun: 3.0 eV
	Disc: NASA01	# of Scans: 1	Resolution: 4

Description: HDPE-21, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN. Operator: WBC
CHARGE NEUTRALIZATION SCREEN



Report #: MSFC-01

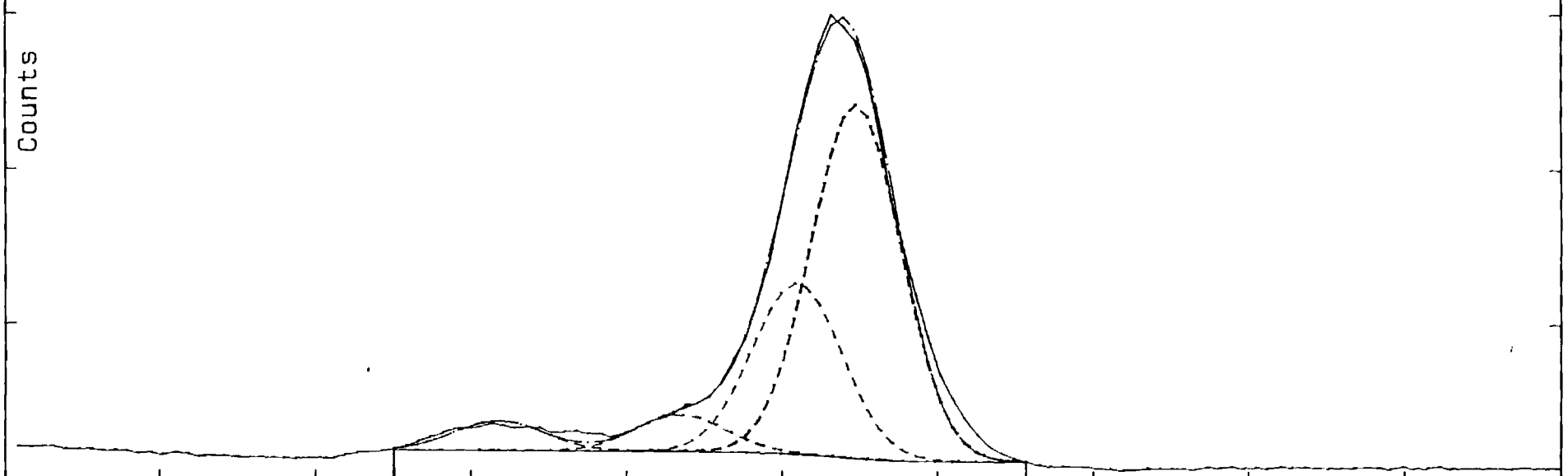
File: HDPEMRS15	Date: 8/15/1988	Spot Size: 300 u	Flood Gun: 3.0 eV
Region 1	Disc: NASA01	# of Scans: 15	Resolution: 2

Description: HDPE-21, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN.; CHARGE NEUTRALIZATION SCREEN

Operator: WBC

10000

Energy	Width	Area	%
279.05	1.35	42041	59.9
279.81	1.35	20234	28.9
281.31	1.35	4345	6.2
283.65	1.35	3511	5.0



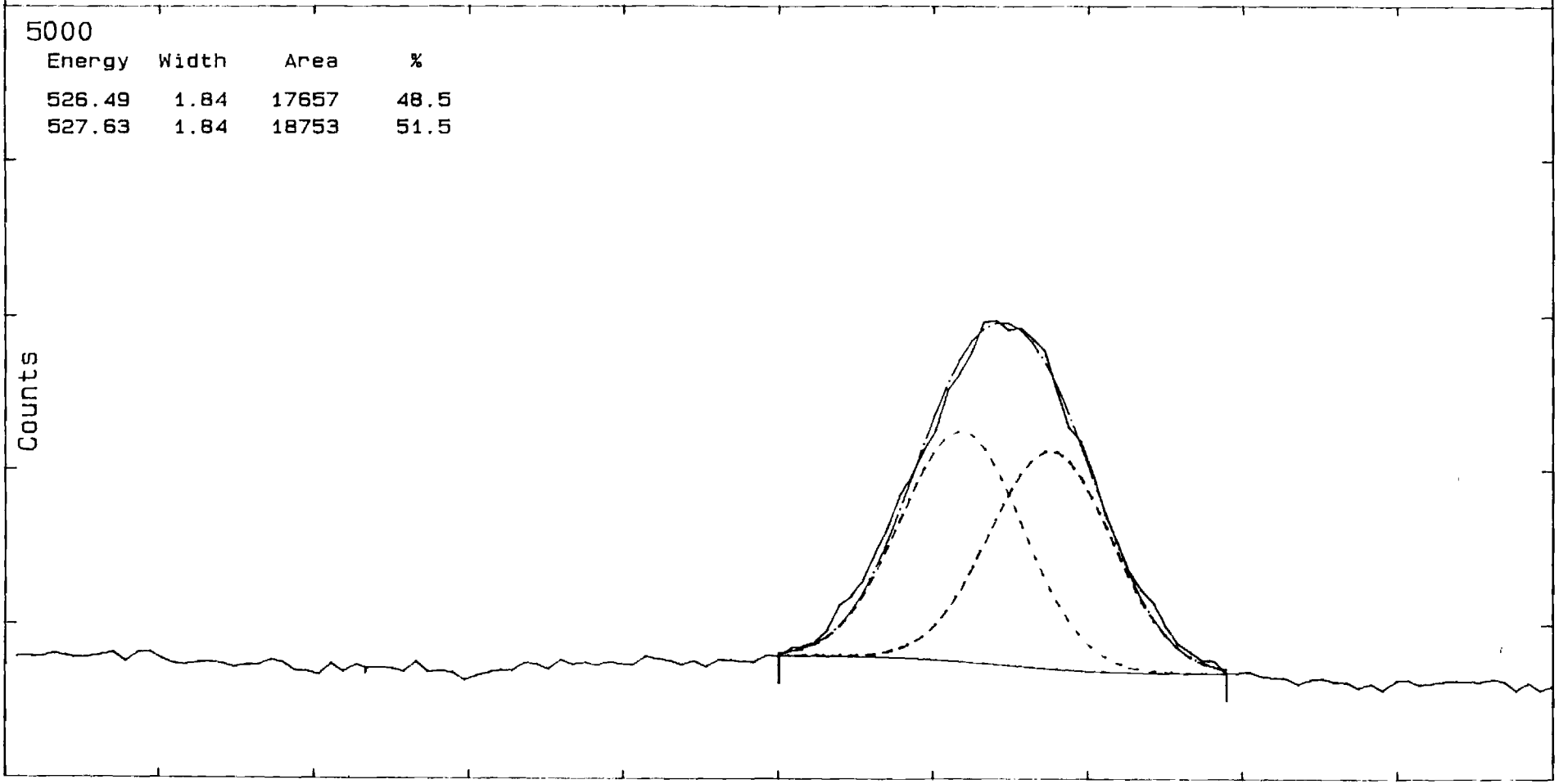
290.0 Binding Energy (eV) 270.0

Report #: MSFC-01

File: HDPEMRS15	Date: 8/15/1988	Spot Size: 300 u	Flood Gun: 3.0 eV
Region 2	Disc: NASA01	# of Scans: 15	Resolution: 2

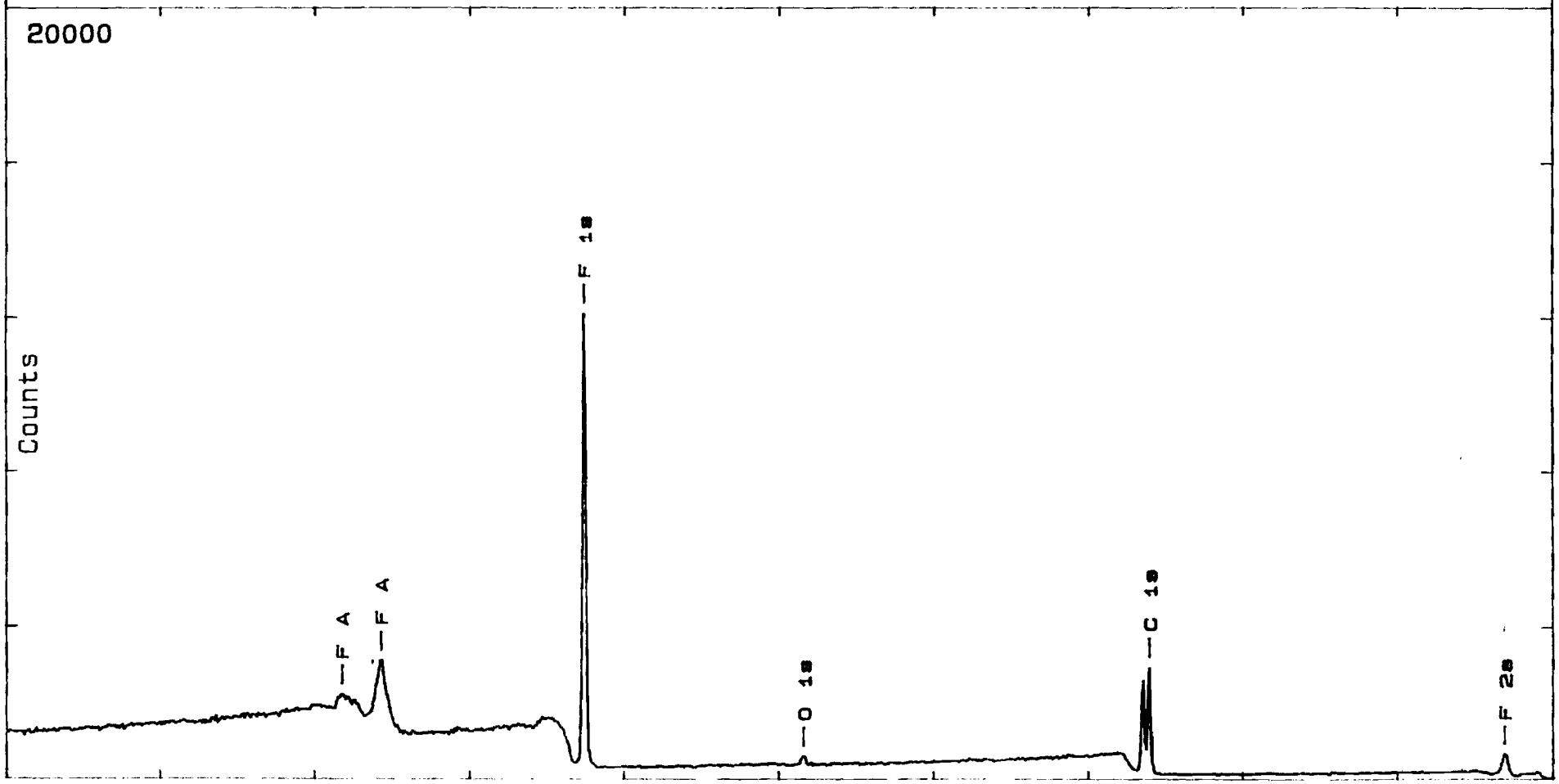
Description: HDPE-21, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN.; CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

File: PVDFMRS08	Date: 8/17/1988	Spot Size: 1000 u	Flood Gun: 7.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4
Description: PVDF-4, CONTROL (NO EXPOSURE) CHARGE NEUTRALIZATION SCREEN			Operator: WBC

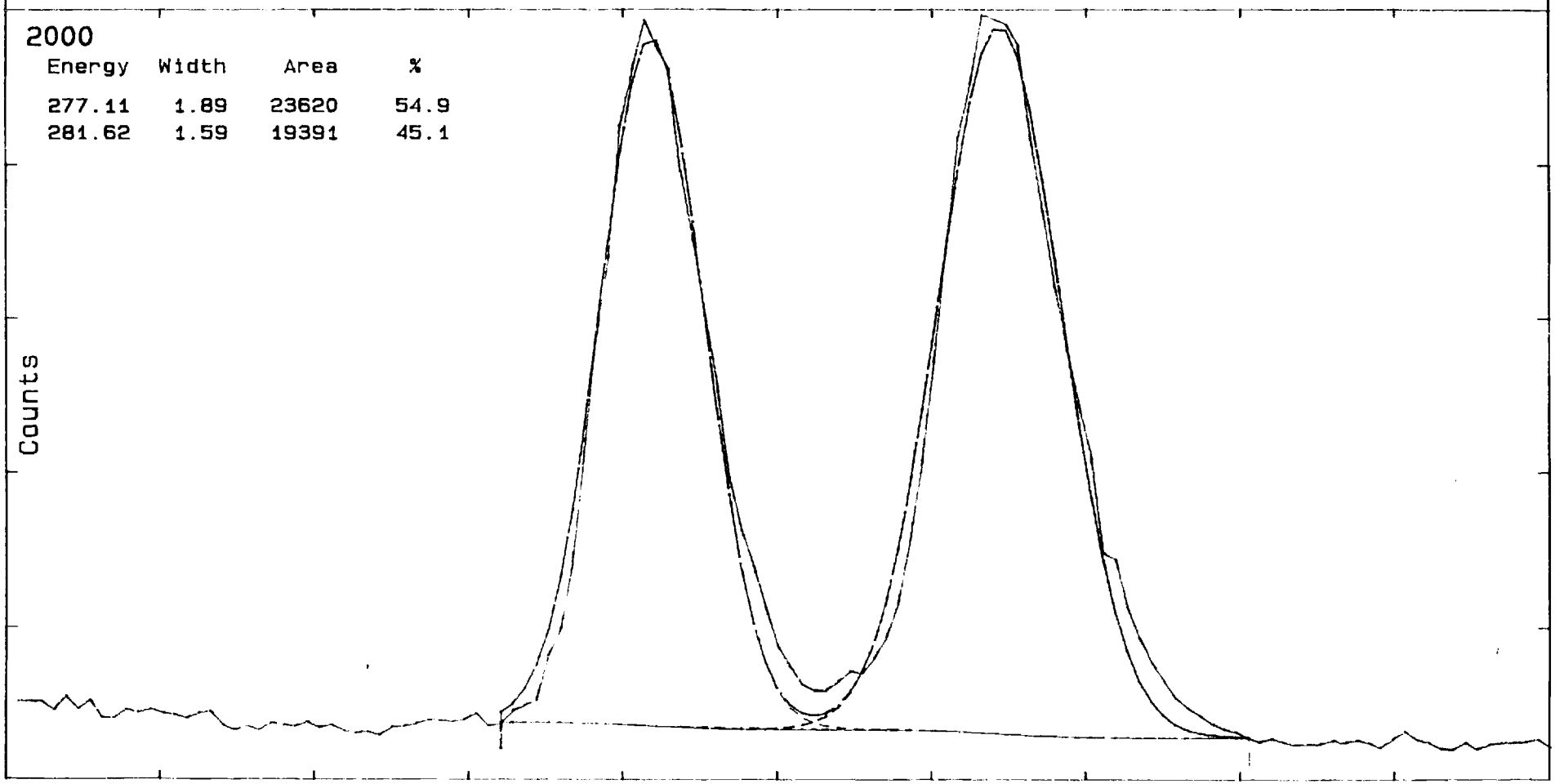


Report #: MSFC-01

File: PVDFMRS08	Date: 8/17/1988	Spot Size: 300 u	Flood Gun: 7.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-4 (CONTROL), C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

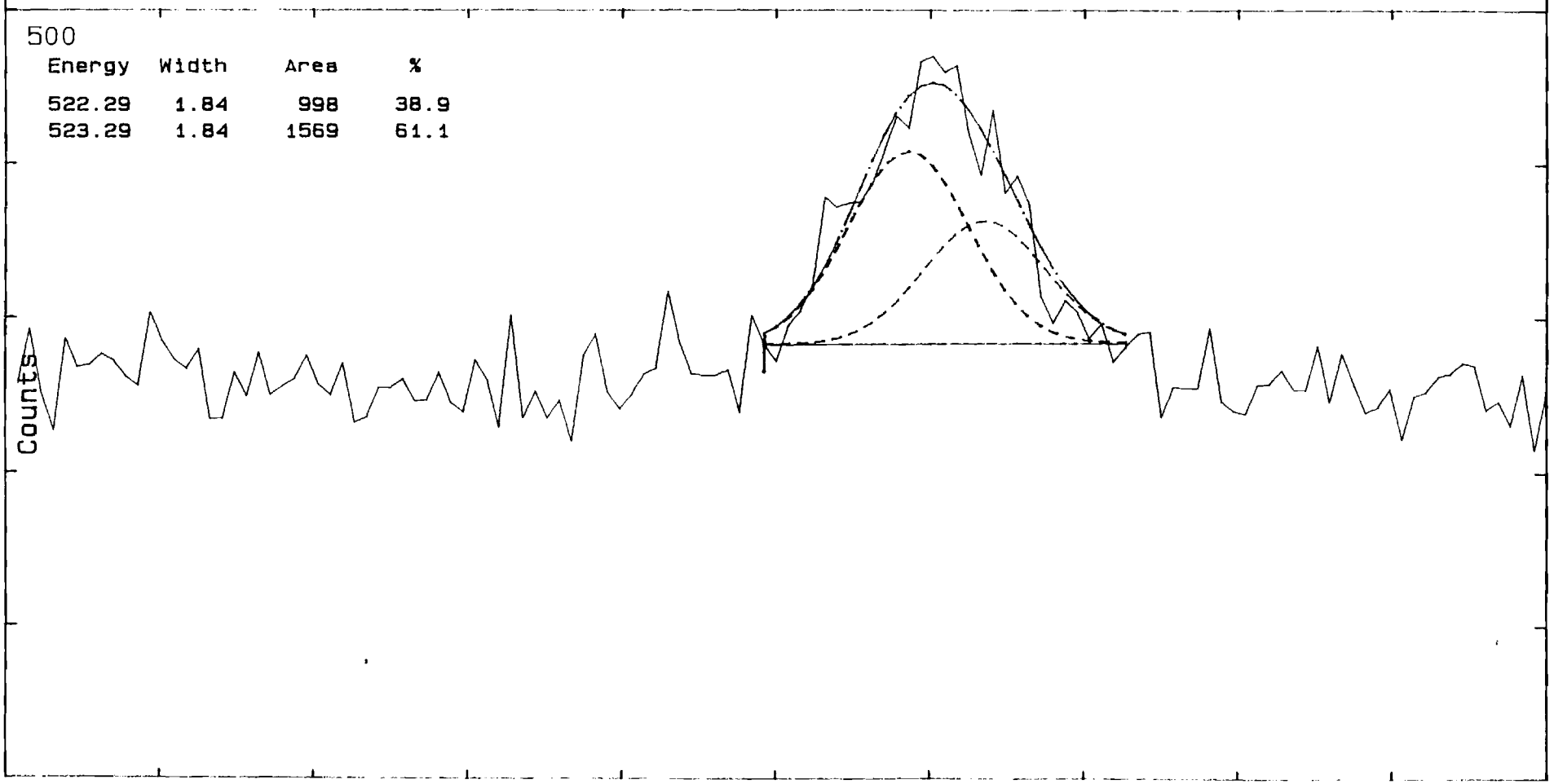


Report #: MSFC-01

File: PVDFMRS08	Date: 8/17/1988	Spot Size: 300 u	Flood Gun: 7.0 eV
Region 3	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-4 (CONTROL), O 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC



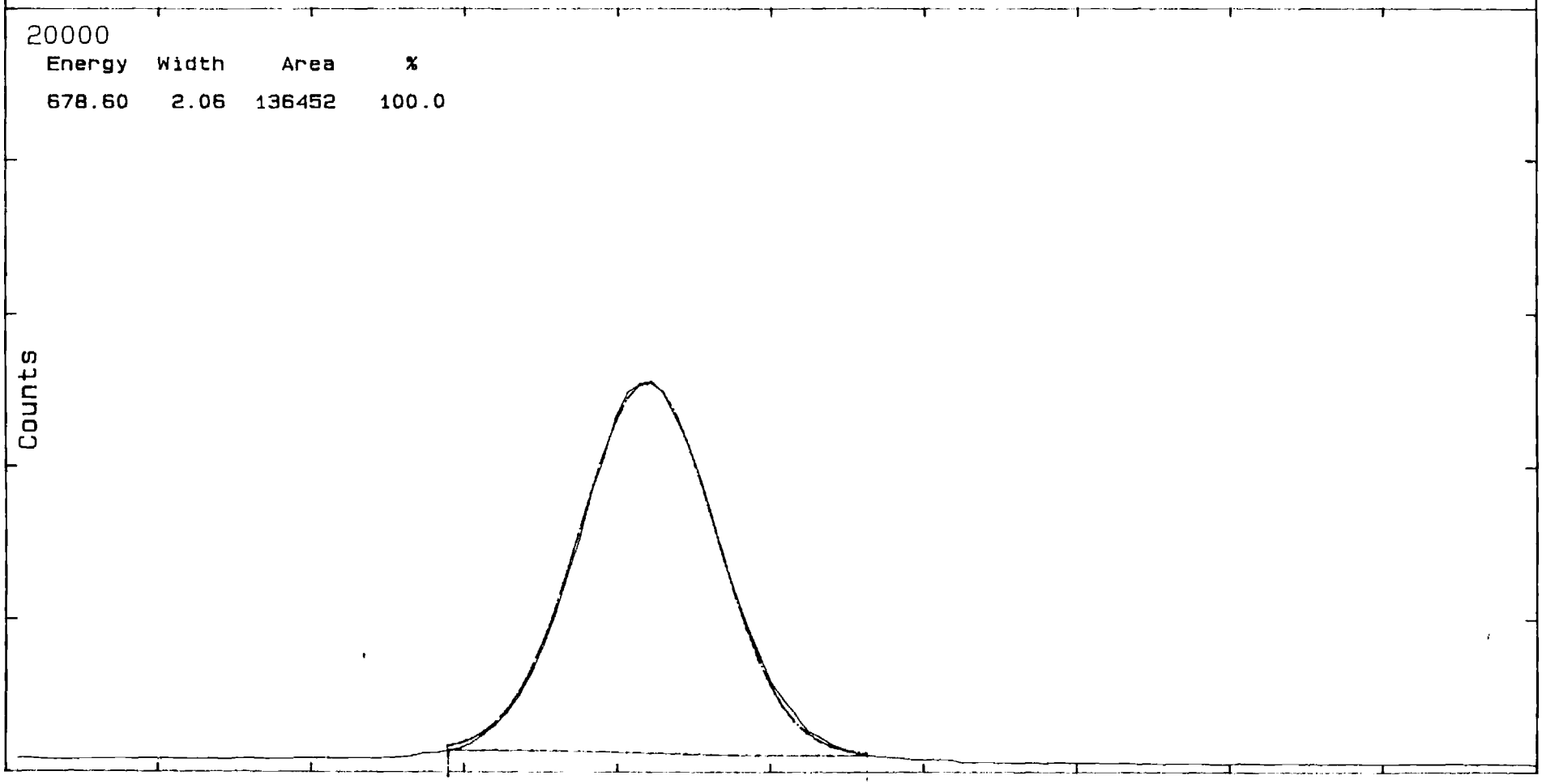
535.0 Binding Energy (eV) 515.0

Report #: MSFC-01

File: PVDFMRS08	Date: 8/17/1988	Spot Size: 300 u	Flood Gun: 7.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-4 (CONTROL), F 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

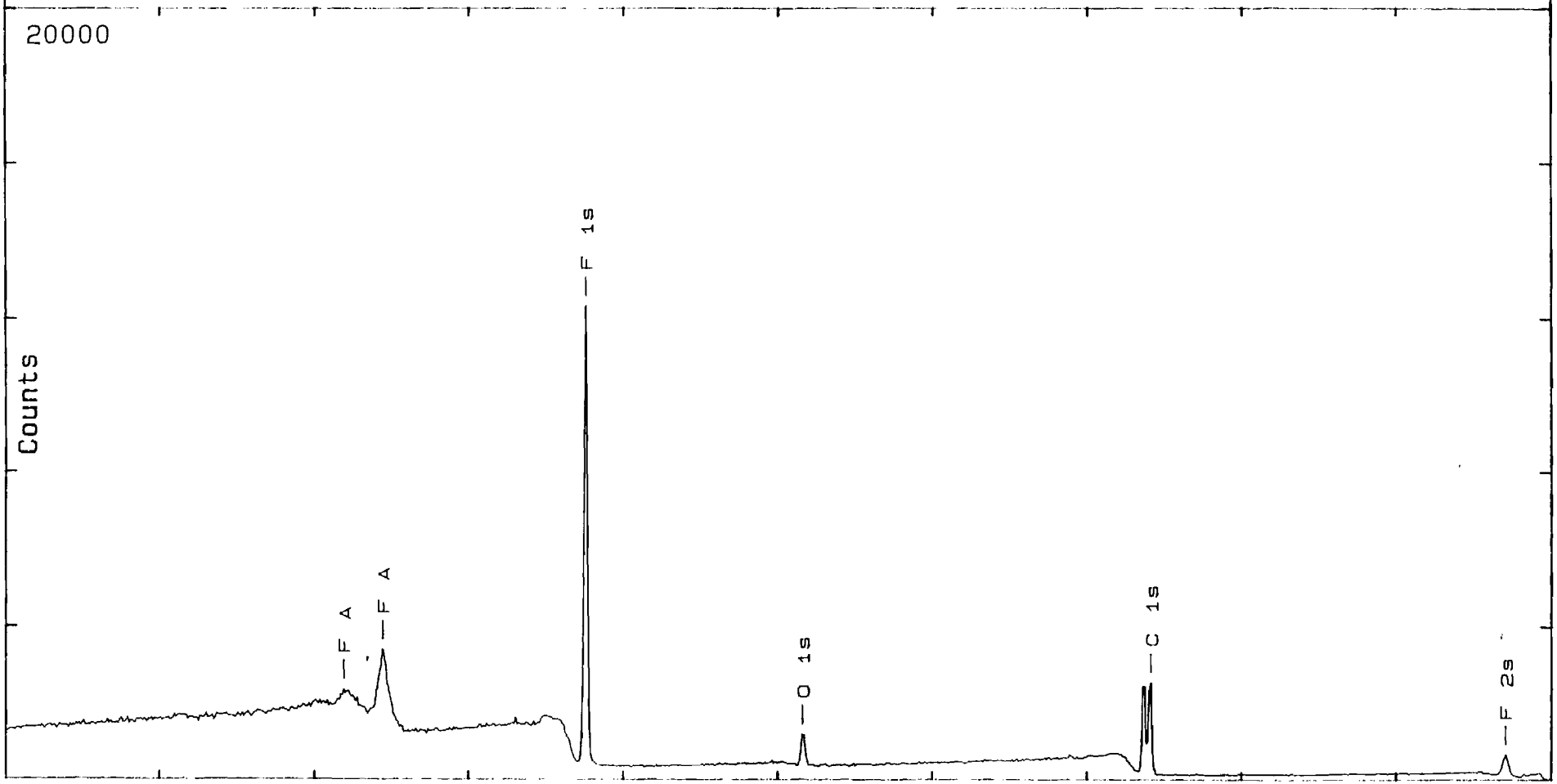


Report #: MSFC-01

File: PVDFMRS09	Date: 8/18/1988	Spot Size: 1000 u	Flood Gun: 9.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4

Description: PVDF-5, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN. CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

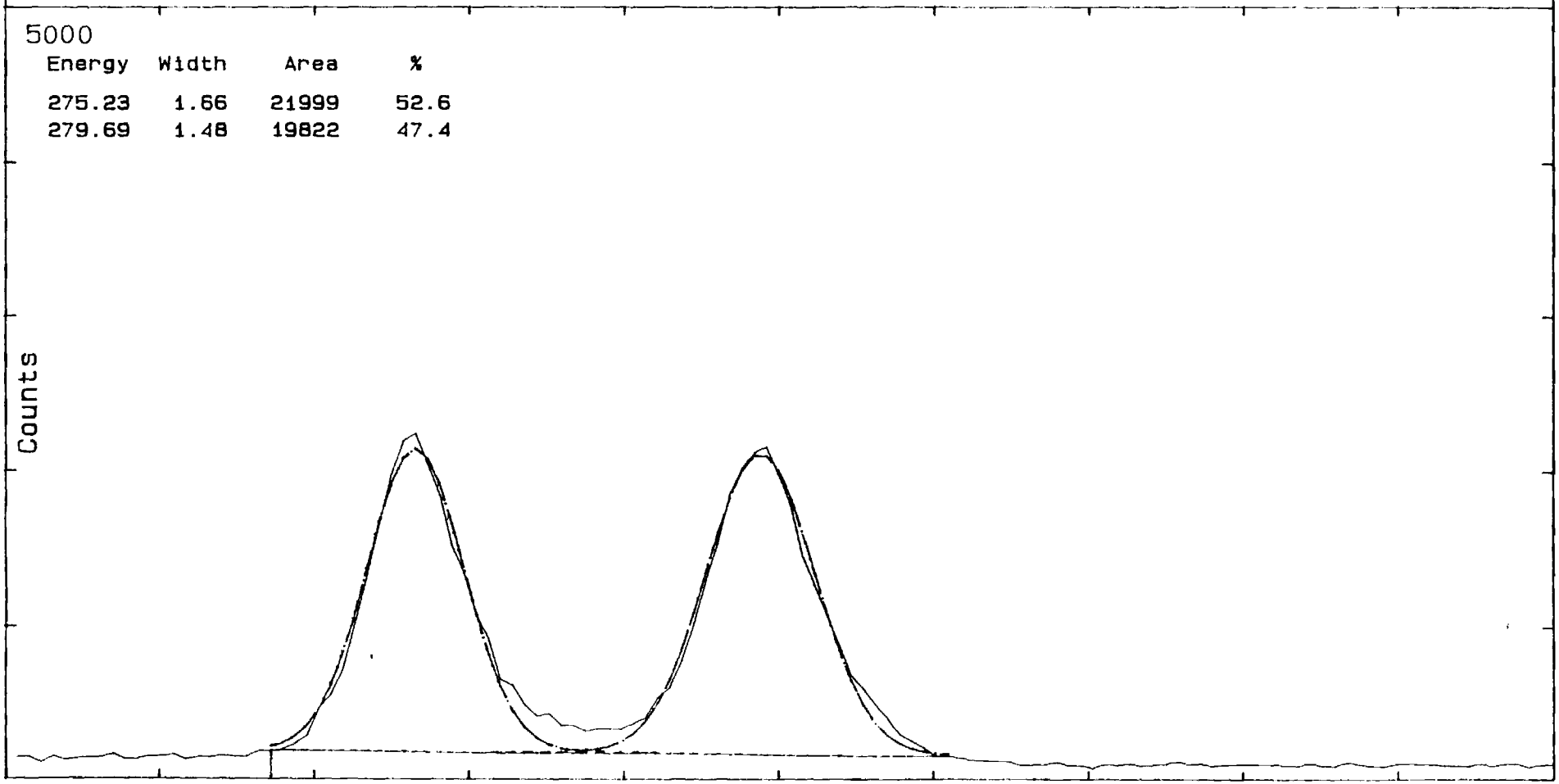
File: PVDFMRS09	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 9.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-5, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

5000

Energy	Width	Area	%
275.23	1.66	21999	52.6
279.69	1.48	19822	47.4



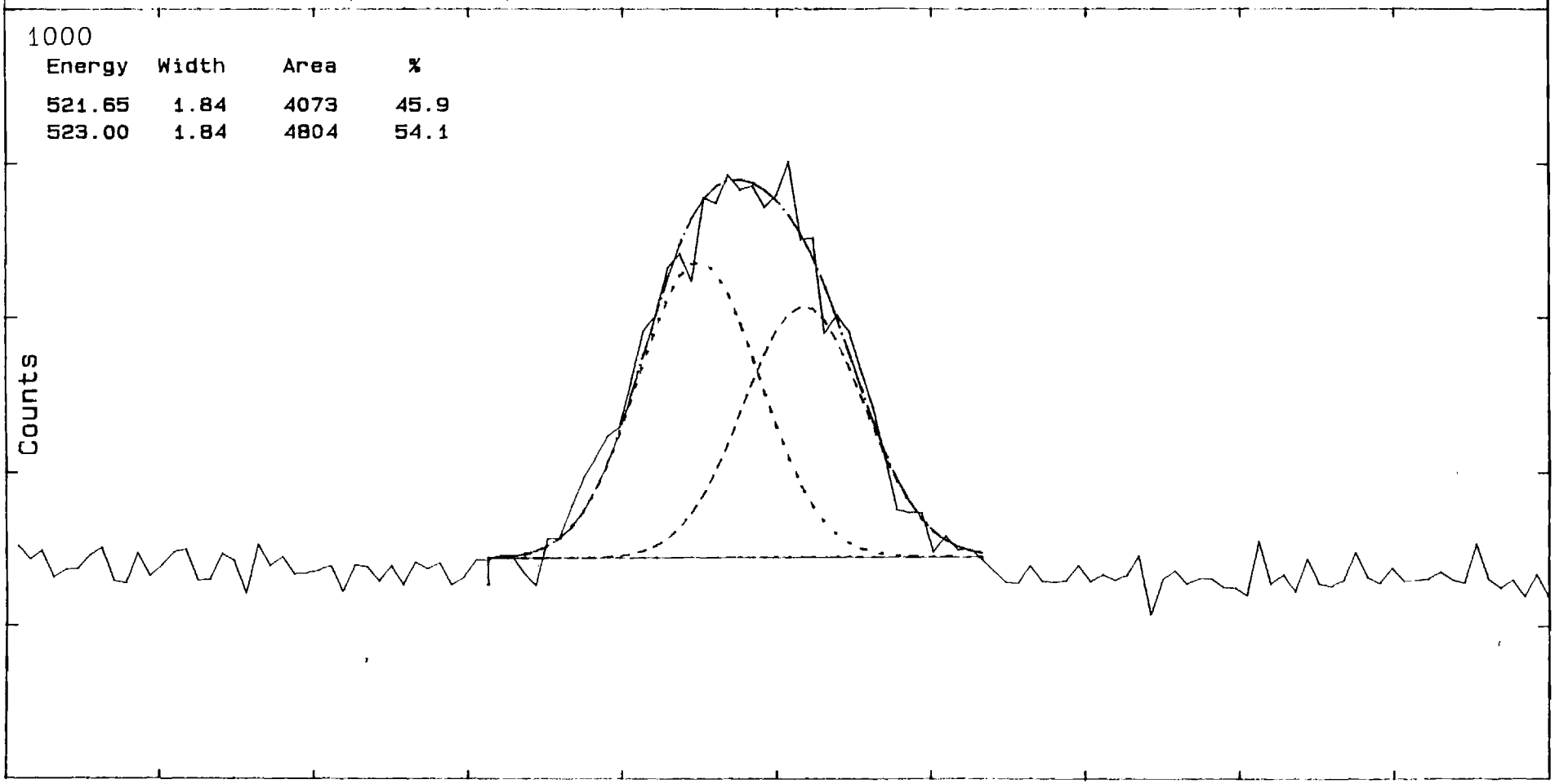
285.0 Binding Energy (eV) 265.0

Report #: MSFC-01

File: PVDFMRS09	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 9.0 eV
Region 3	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-5, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

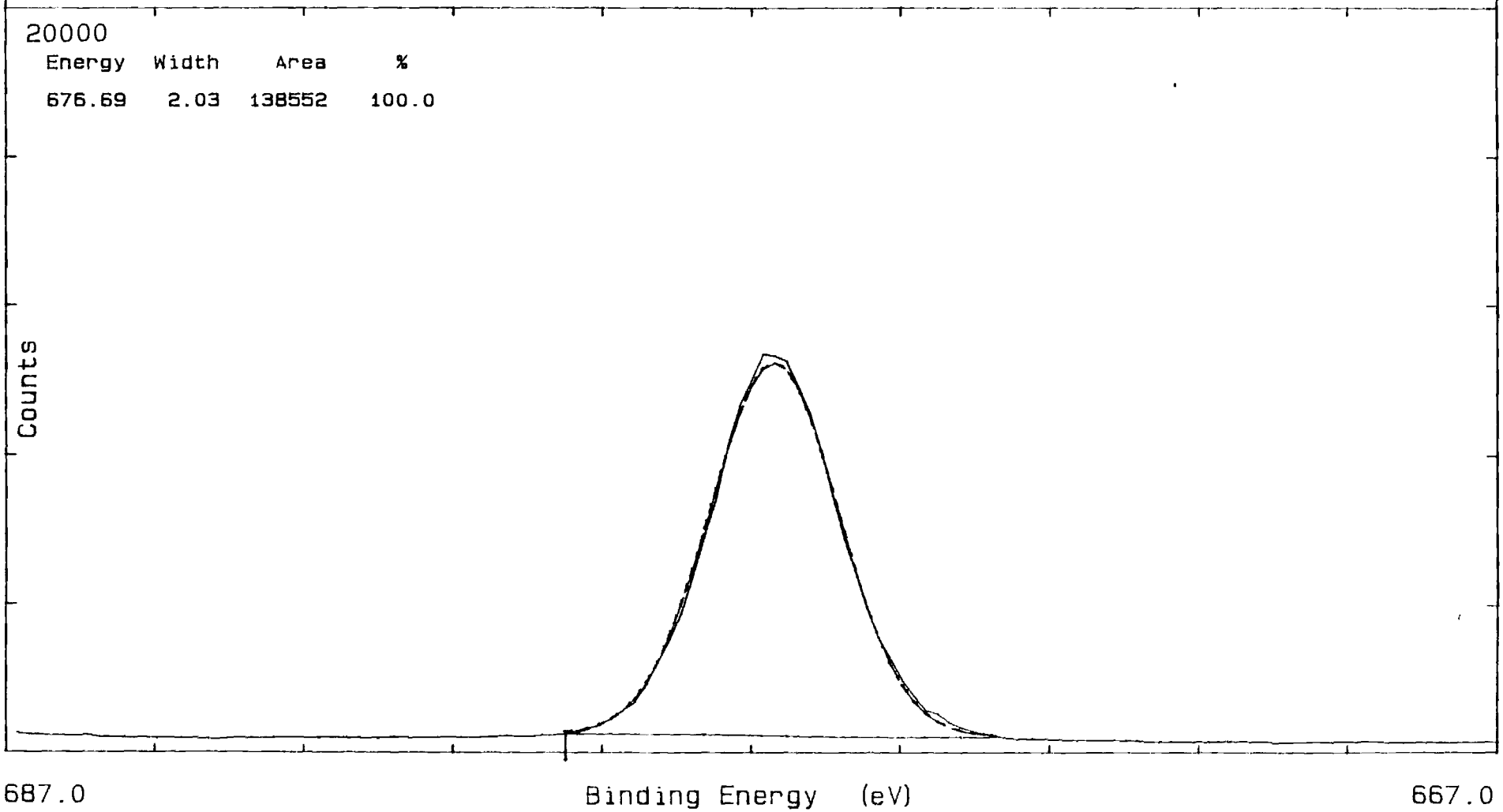


Report #: MSFC-01

File: PVDFMRS09	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 9.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2

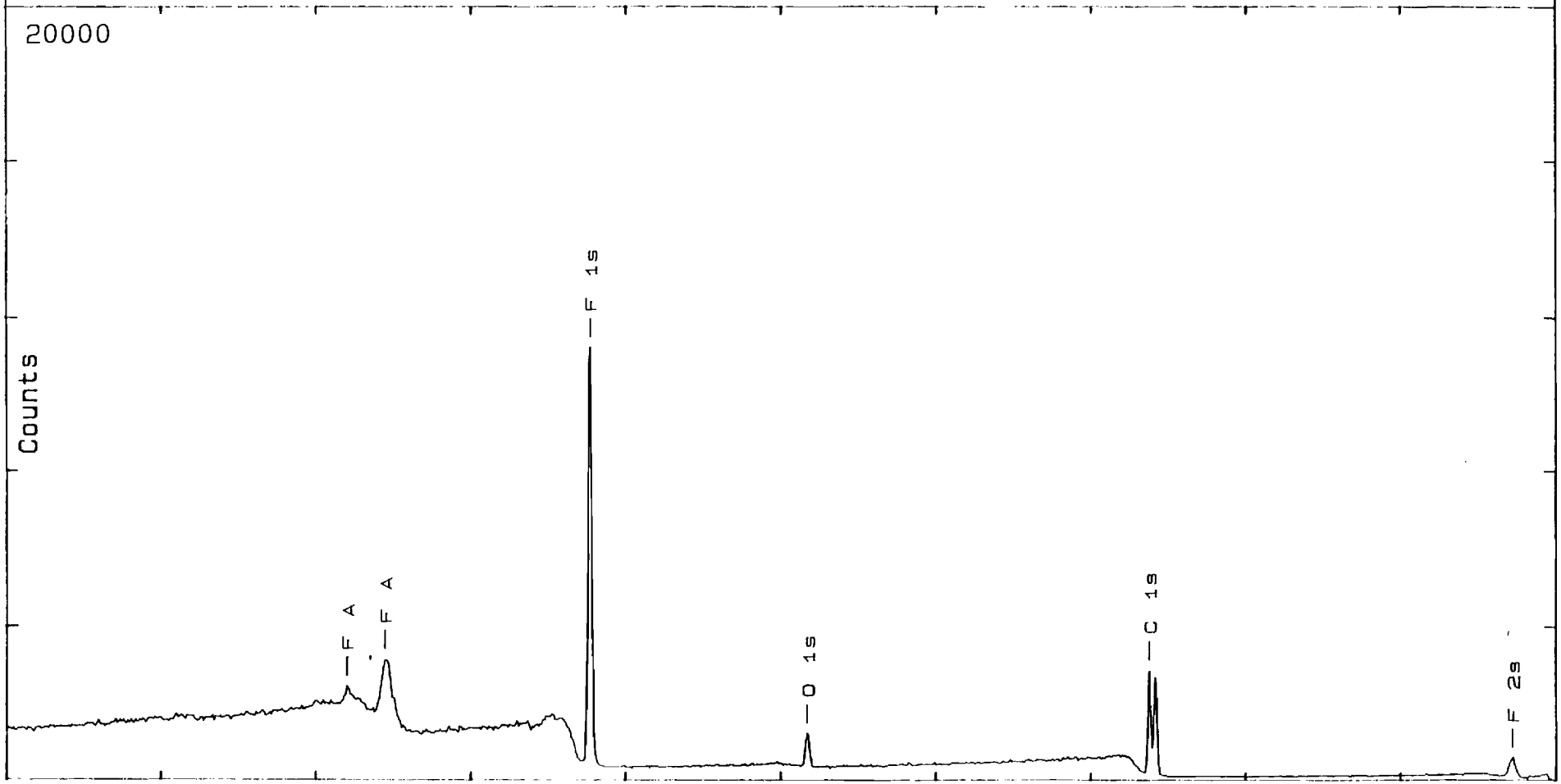
Description: PVDF-5, F 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 30 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

File: PVDFMRS10	Date: 8/18/1988	Spot Size: 1000 u	Flood Gun: 11.0 eV
Region 1	Disc: NASA01	# of Scans: 1	Resolution: 4
Description: PVDF-6, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN. CHARGE NEUTRALIZATION SCREEN			Operator: WBC

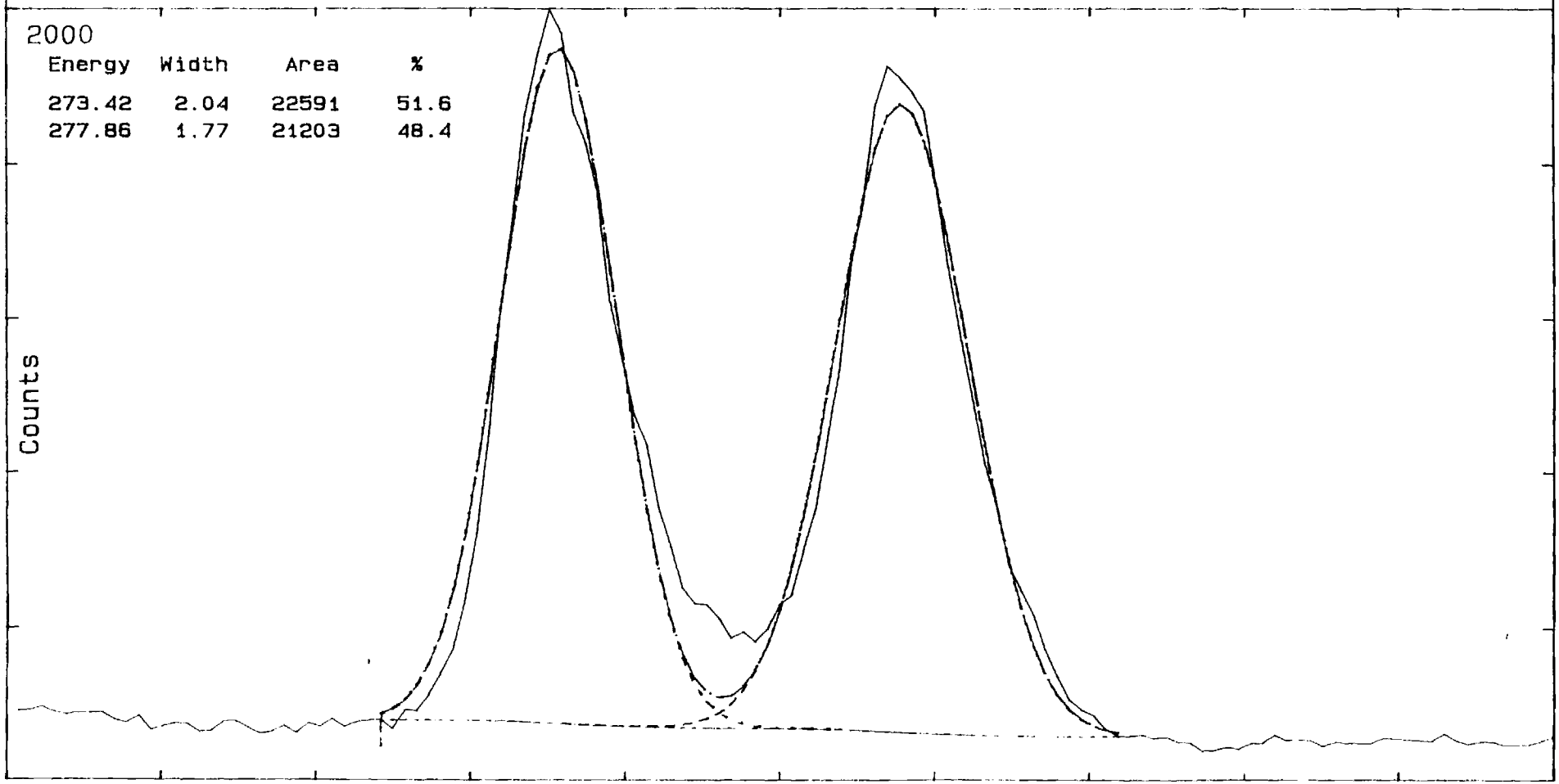


Report #: MSFC-01

File: PVDFMRS10	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 11.0 eV
Region 2	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-6, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

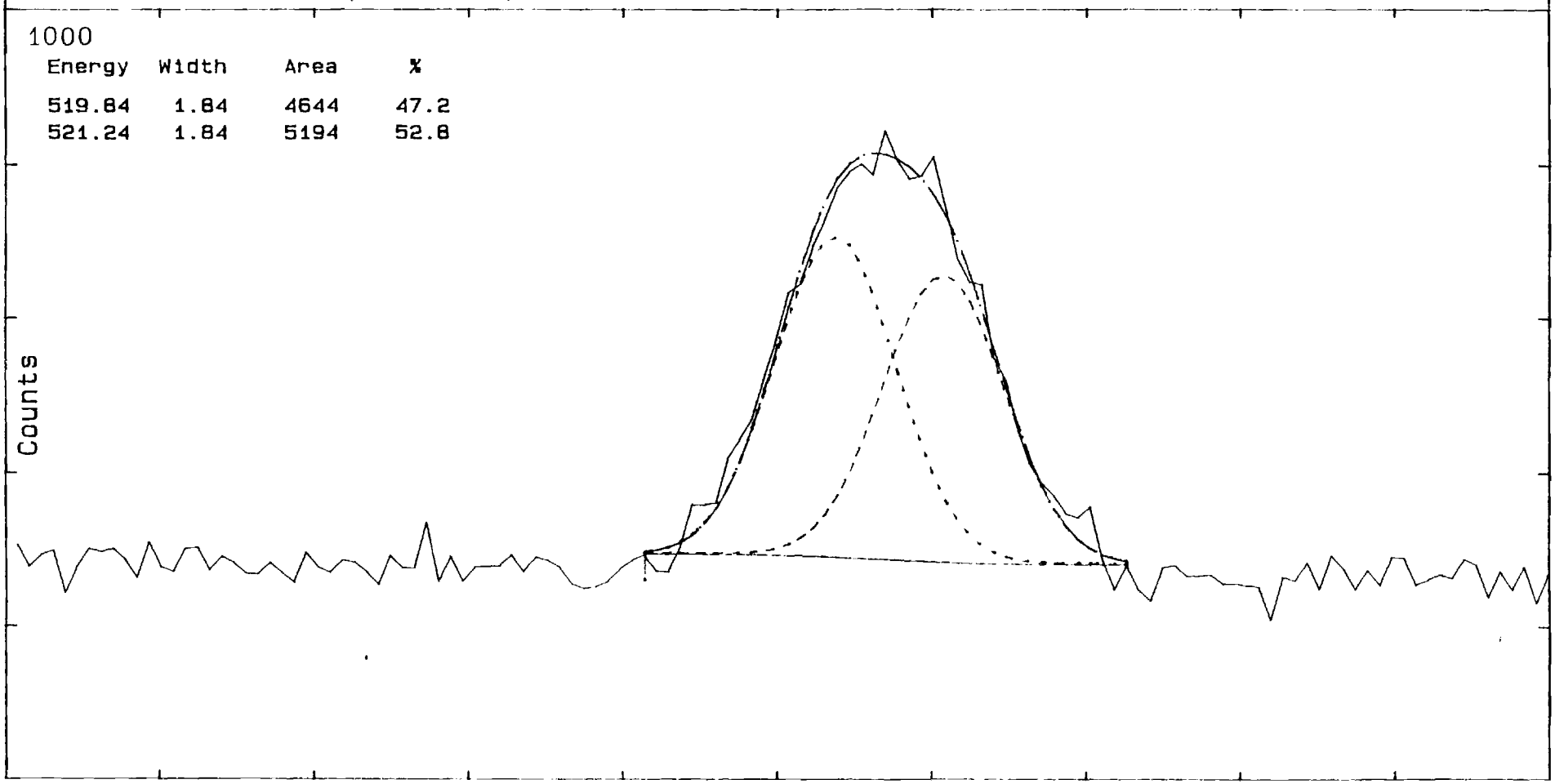


Report #: MSFC-01

File: PVDFMRS10	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 11.0 eV
Region 3	Disc: NASA01	# of Scans: 10	Resolution: 2

Description: PVDF-6, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

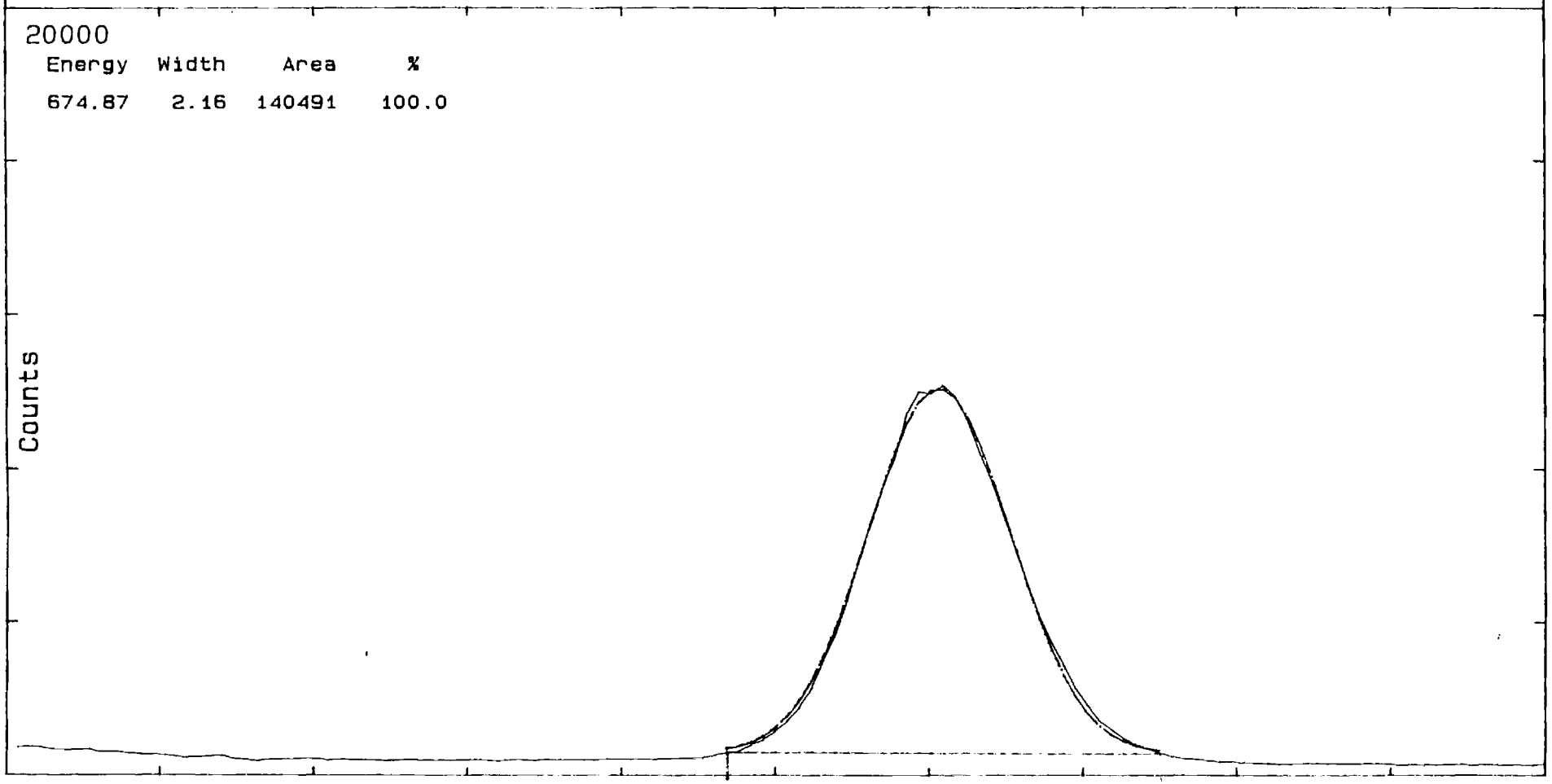


Report #: MSFC-01

File: PVDFMRS10	Date: 8/18/1988	Spot Size: 300 u	Flood Gun: 11.0 eV
Region 4	Disc: NASA01	# of Scans: 10	Resolution: 2

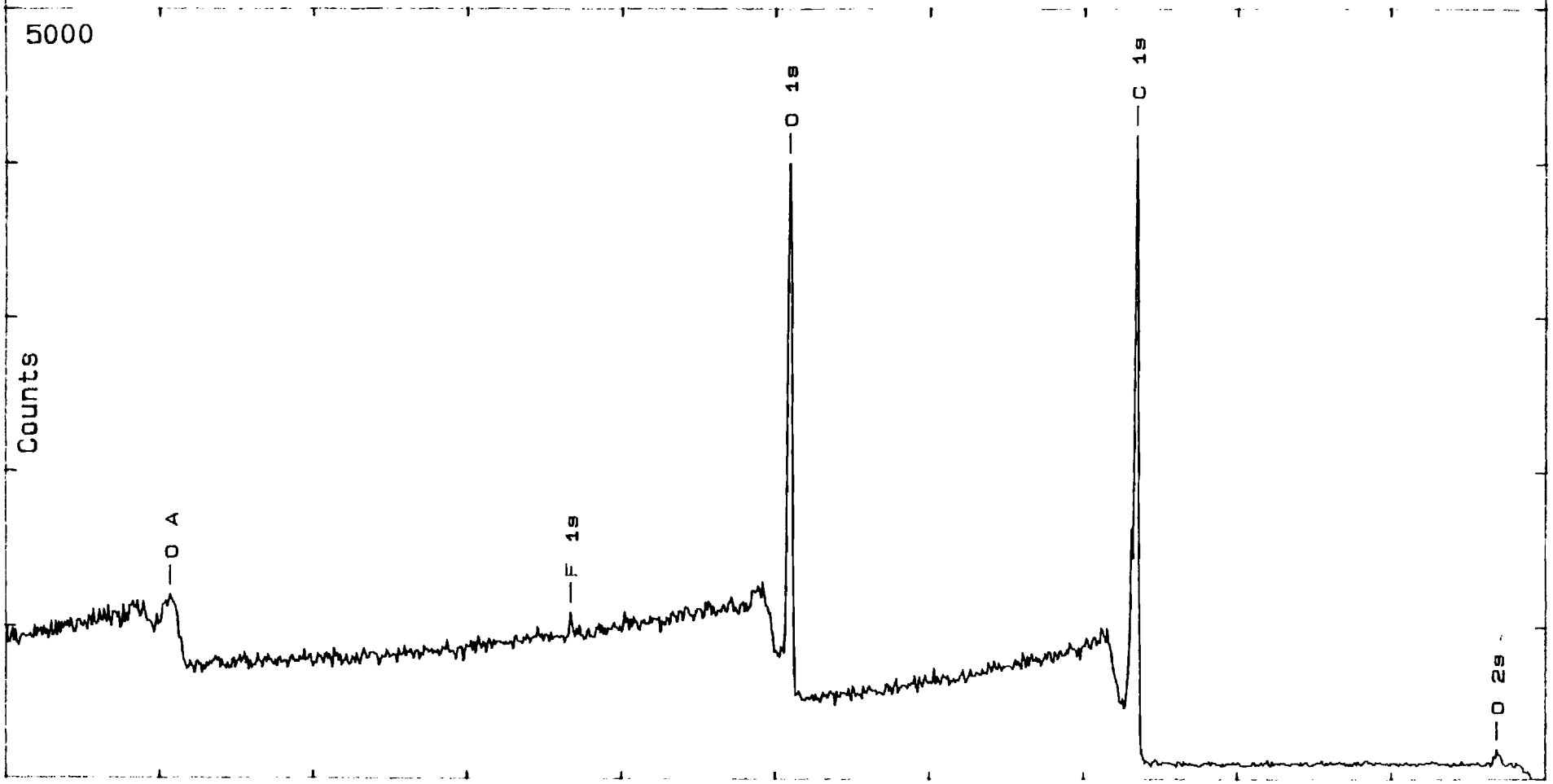
Description: PVDF-6, F 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 95 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-01

File: MYLAR01	Date: 8/25/1988	Spot Size: 1000 u	Flood Gun: 1.0 eV
	Disc: NASA02	# of Scans: 1	Resolution: 4
Description: MYLAR CONTROL CHARGE NEUTRALIZATION SCREEN			Operator: WBC



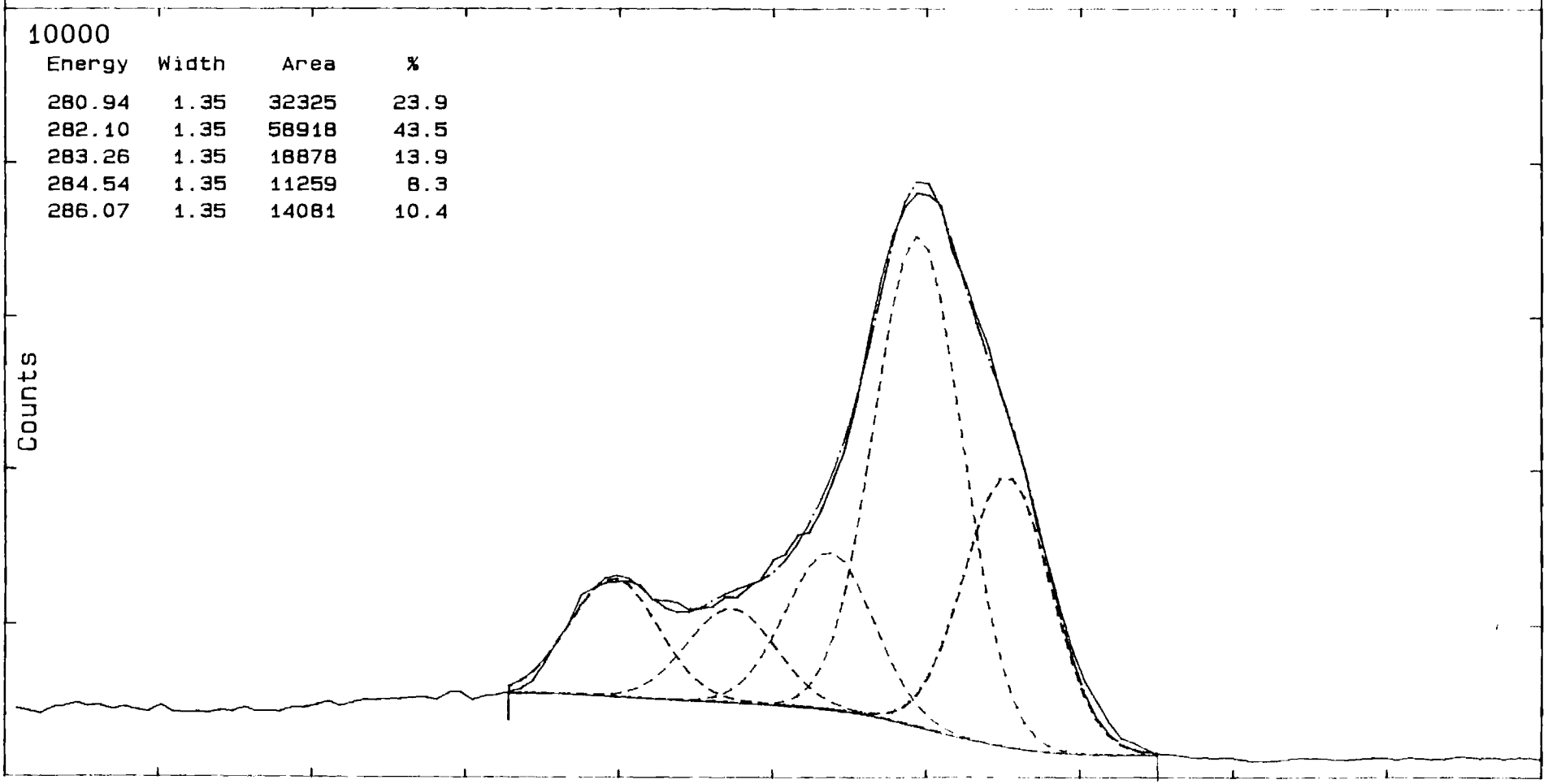
1090.0 Binding Energy (eV) -10.0

Report #: MSFC-02

File: MYLARMRS01	Date: 8/25/1988	Spot Size: 300 u	Flood Gun: 1.0 eV
Region 1	Disc: NASA02	# of Scans: 30	Resolution: 2

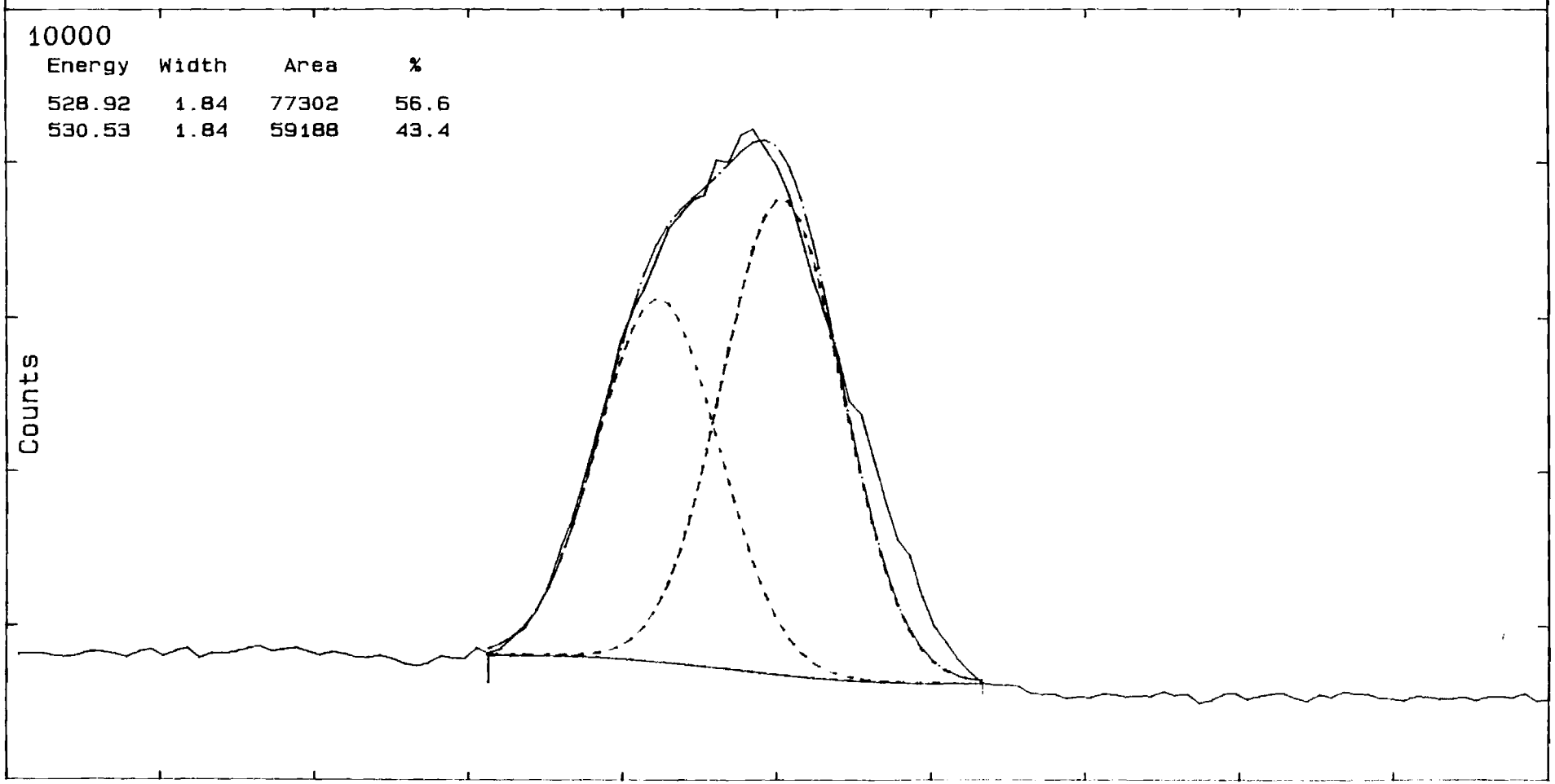
Description: MYLAR CONTROL, C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC



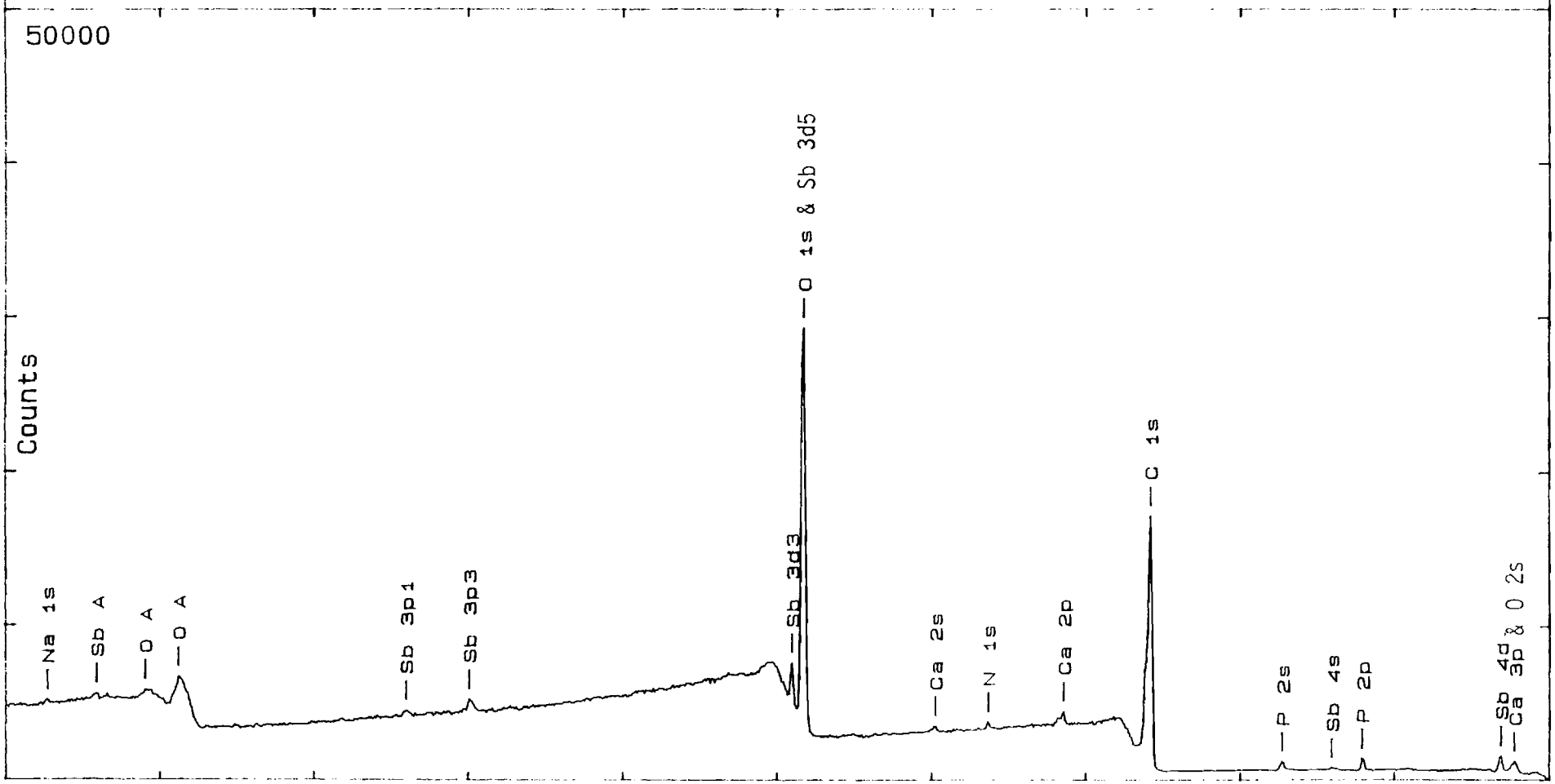
Report #: MSFC-02

File: MYLARMRS01	Date: 8/25/1988	Spot Size: 300 u	Flood Gun: 1.0 eV
Region 2	Disc: NASA02	# of Scans: 30	Resolution: 2
Description: MYLAR CONTROL, O 1s SPECTRUM CHARGE NEUTRALIZATION SCREEN			Operator: WBC



Report #: MSFC-02

File: MYLAR02	Date: 8/25/1988	Spot Size: 1000 u	Flood Gun: 9.0 eV
	Disc: NASA02	# of Scans: 5	Resolution: 4
Description: MYLAR-11, PLASMA EXPOSED: 45 DEG C, 60 W, 75 MIN. CHARGE NEUTRALIZATION SCREEN			Operator: WBC



1090.0 Binding Energy (eV) -10.0

Report #: MSFC-02

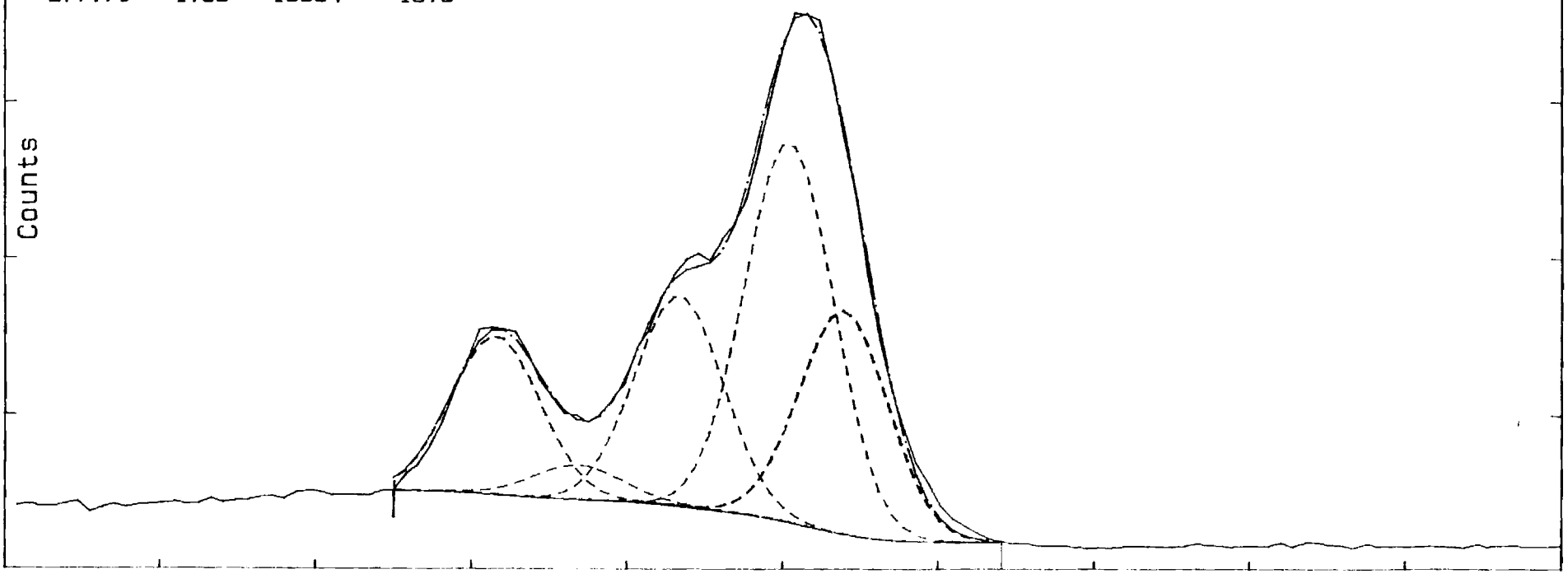
File: MYLARMRS03	Date: 8/25/1988	Spot Size: 300 u	Flood Gun: 9.0 eV
Region 1	Disc: NASA02	# of Scans: 30	Resolution: 2

Description: MYLAR-11, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 75 MIN., CHARGE NEUTRALIZATION SCREEN

Operator: WBC

10000

Energy	Width	Area	%
273.21	1.35	26557	22.2
273.91	1.35	44954	37.6
275.33	1.35	24984	20.9
276.64	1.35	4092	3.4
277.70	1.35	18854	15.8



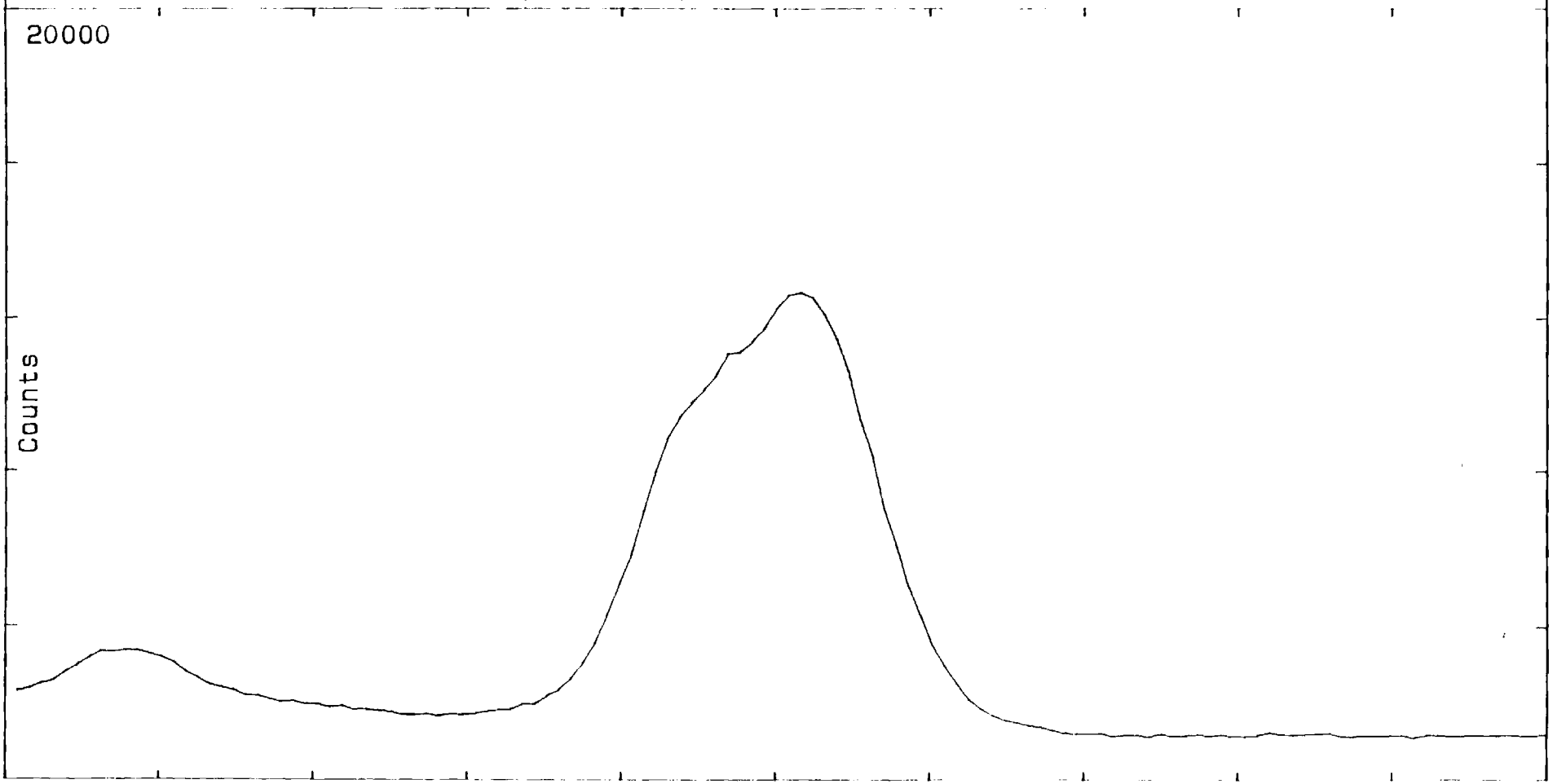
284.0 Binding Energy (eV) 264.0

Report #: MSFC-02

File: MYLARMRS03	Date: 8/25/1988	Spot Size: 300 u	Flood Gun: 9.0 eV
Region 2	Disc: NASA02	# of Scans: 30	Resolution: 2

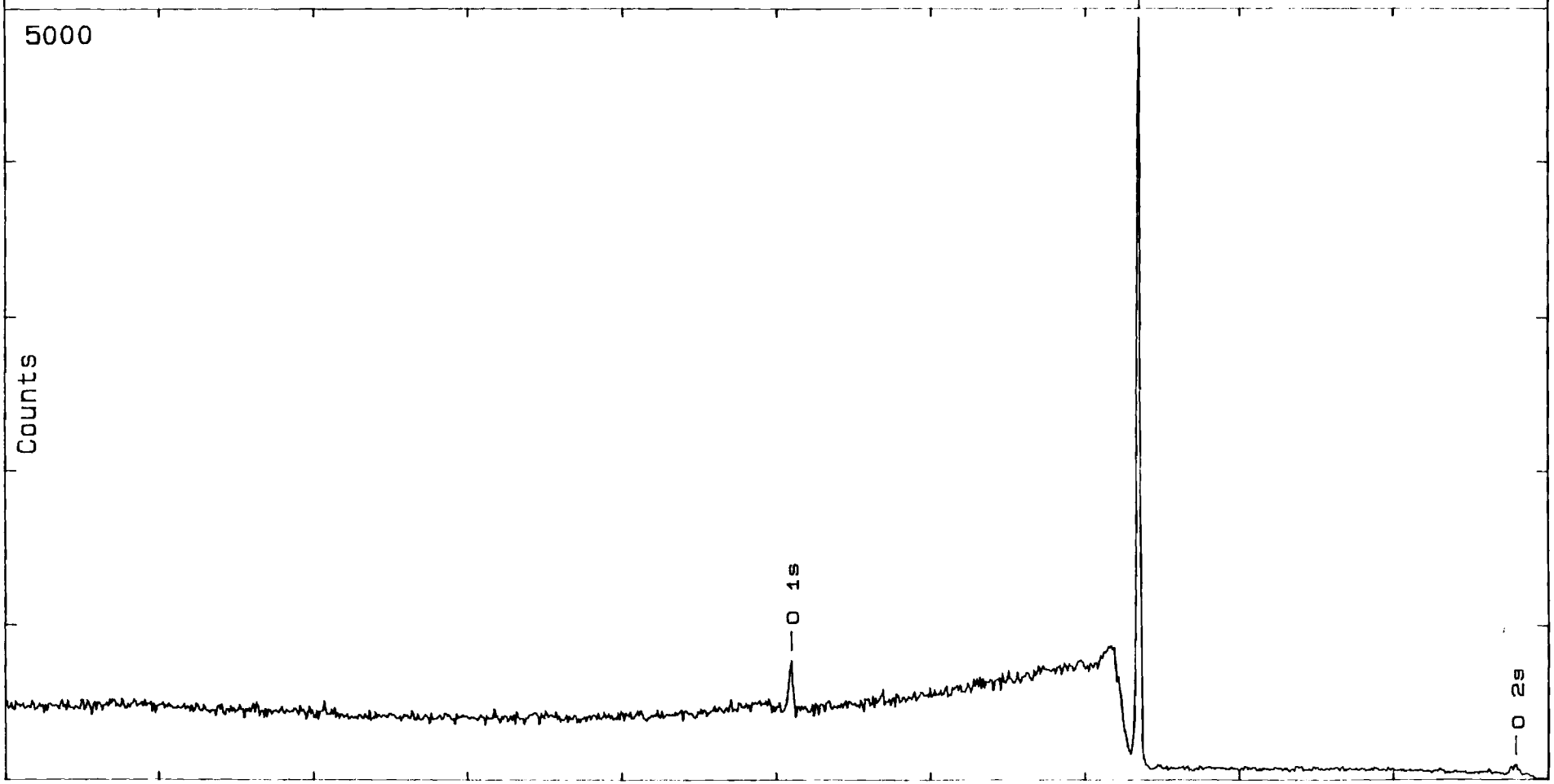
Description: MYLAR-11, O 1s & Sb 3d SPECTRUM, PLASMA EXPOSED: 45 DEG C, 60 W, 75 MIN., NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-02

File: PPROPMRS01	Date: 8/26/1988	Spot Size: 1000 u	Flood Gun: On, 0 eV
Region 1	Disc: NASA02	# of Scans: 1	Resolution: 4
Description: POLYPROPYLENE CONTROL CHARGE NEUTRALIZATION SCREEN			Operator: WBC

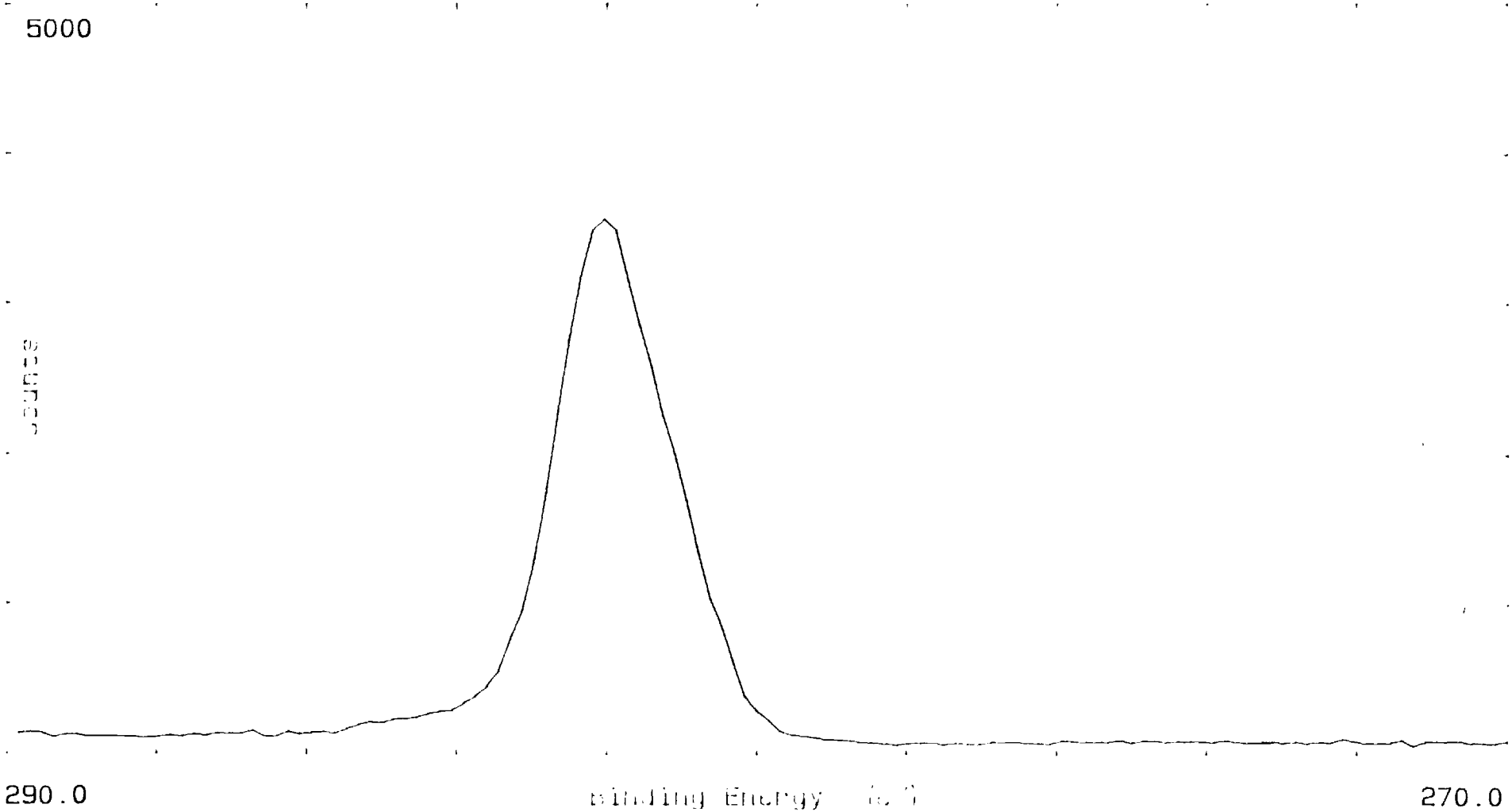


Report #: MSFC-02

File:	PPROPMRS01	Date:	8/26/1988	Spot Size:	300 u	Neutralizer On,	0 eV
Region 2		Disk:	NASA02	# of Scans:	10	Resolution:	2

Description: POLYPROPYLENE CONTROL, C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

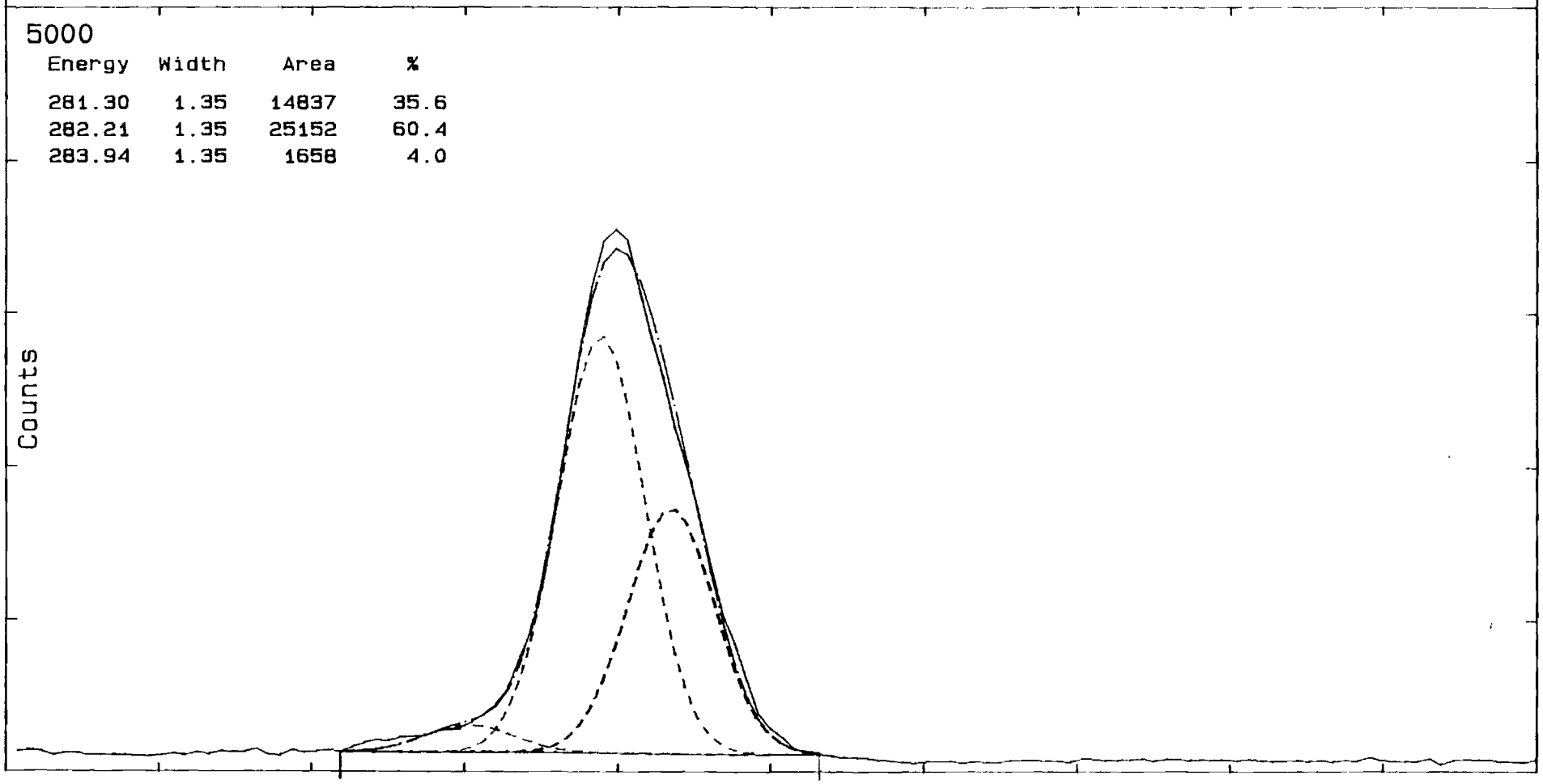


Sample #: MSFC-02

File: PPROPMRS01	Date: 8/26/1988	Spot Size: 300 u	Flood Gun: On, 0 eV
Region 2	Disc: NASA02	# of Scans: 10	Resolution: 2

Description: POLYPROPYLENE CONTROL, C 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC

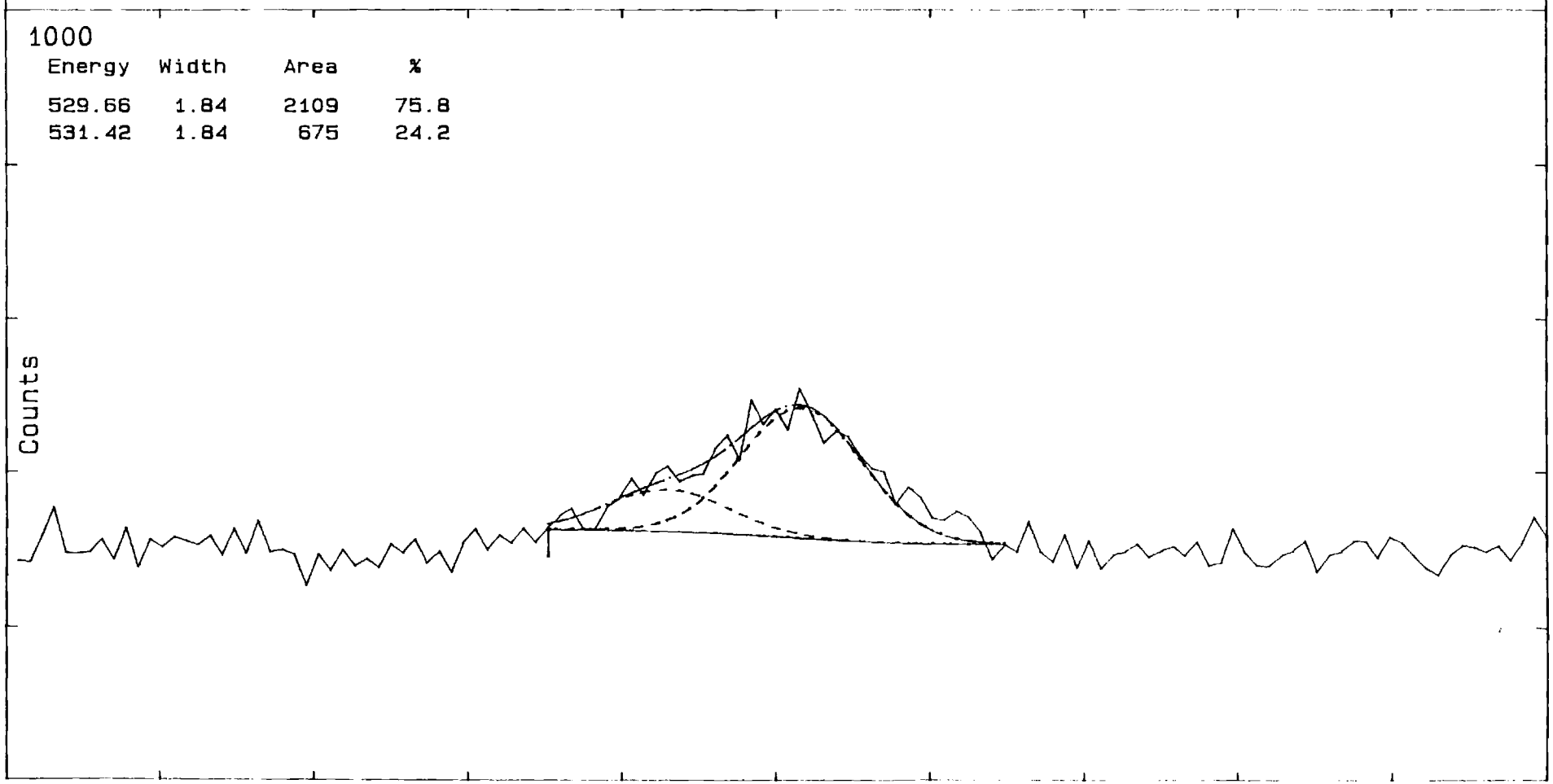


Report #: MSFC-02

File: PPROPMRS01	Date: 8/26/1988	Spot Size: 300 u	Flood Gun: On, 0 eV
Region 3	Disc: NASA02	# of Scans: 10	Resolution: 2

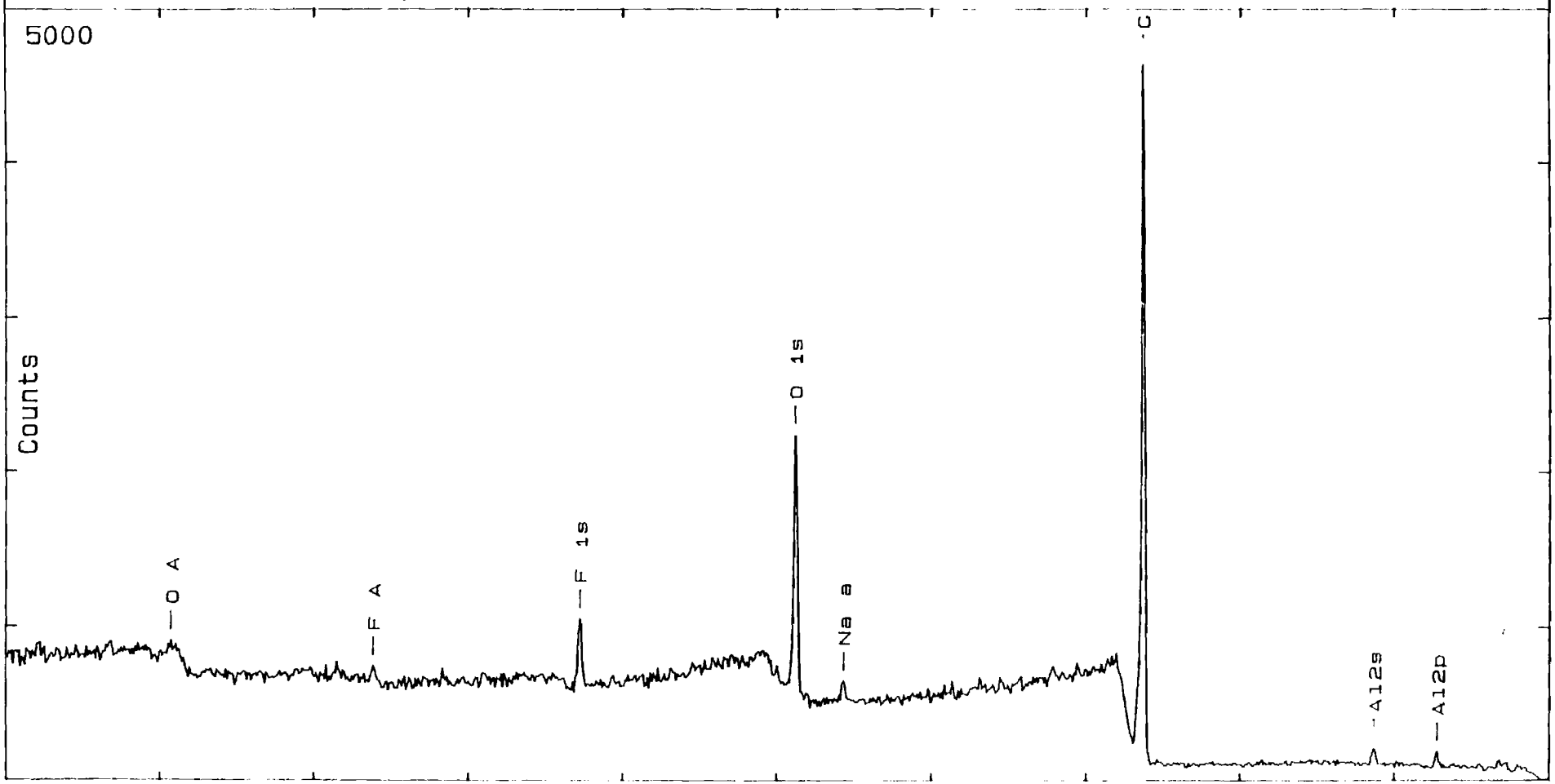
Description: POLYPROPYLENE CONTROL, O 1s SPECTRUM
CHARGE NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-02

File: PPROPMRS02	Date: 8/26/1988	Spot Size: 1000 u	Flood Gun: 3.0 eV
Region 1	Disc: NASA02	# of Scans: 1	Resolution: 4
Description: POLYPROPYLENE-11, PLASMA EXPOSED: 45 DEG C, 100 W, 90 MIN., CHARGE NEUTRALIZATION SCREEN			Operator: WBC



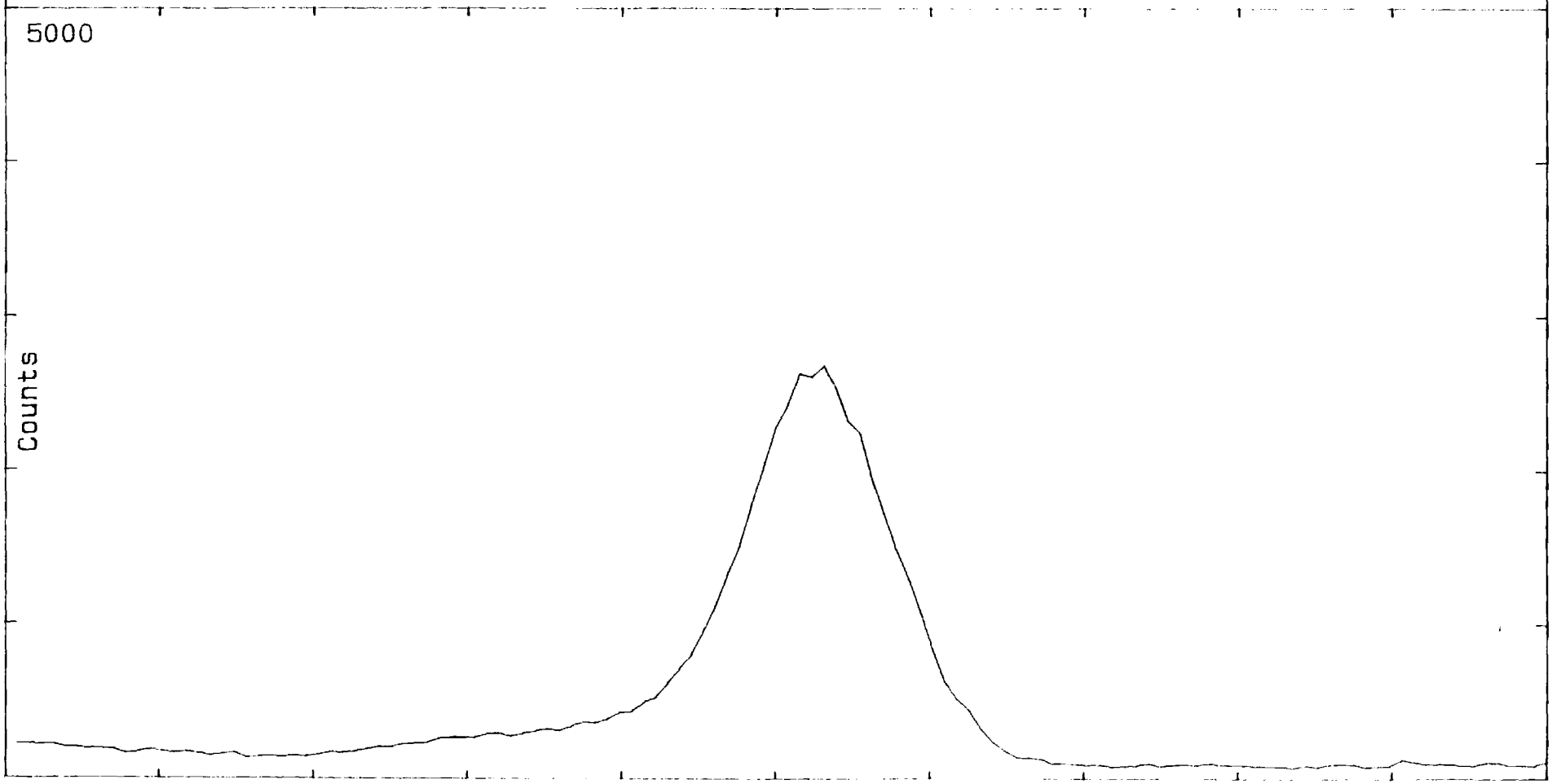
1090.0 Binding Energy (eV) -10.0

Report #: MSFC-02

File: PPROPMRS02	Date: 8/26/1988	Spot Size: 300 u	Flood Gun: 3.0 eV
Region 2	Disc: NASA02	# of Scans: 10	Resolution: 2

Description: POLYPROPYLENE-11, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 100 W, 90 MIN., NEUTRALIZATION SCREEN

Operator: WBC

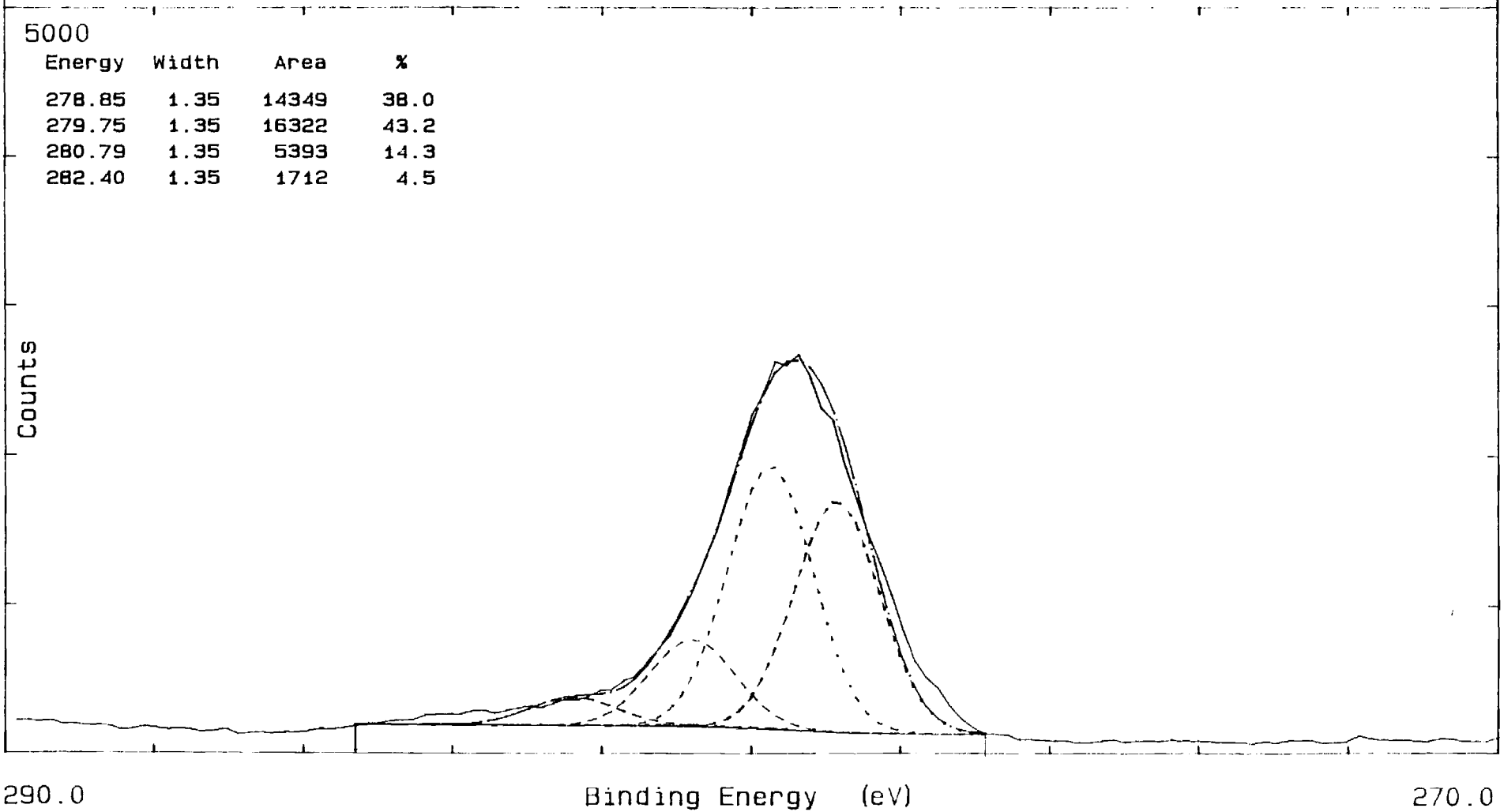


Report #: MSFC-02

File: PPROPMRS02	Date: 8/26/1988	Spot Size: 300 u	Flood Gun: 3.0 eV
Region 2	Disc: NASA02	# of Scans: 10	Resolution: 2

Description: POLYPROPYLENE-11, C 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 100 W, 90 MIN., NEUTRALIZATION SCREEN

Operator: WBC



Report #: MSFC-02

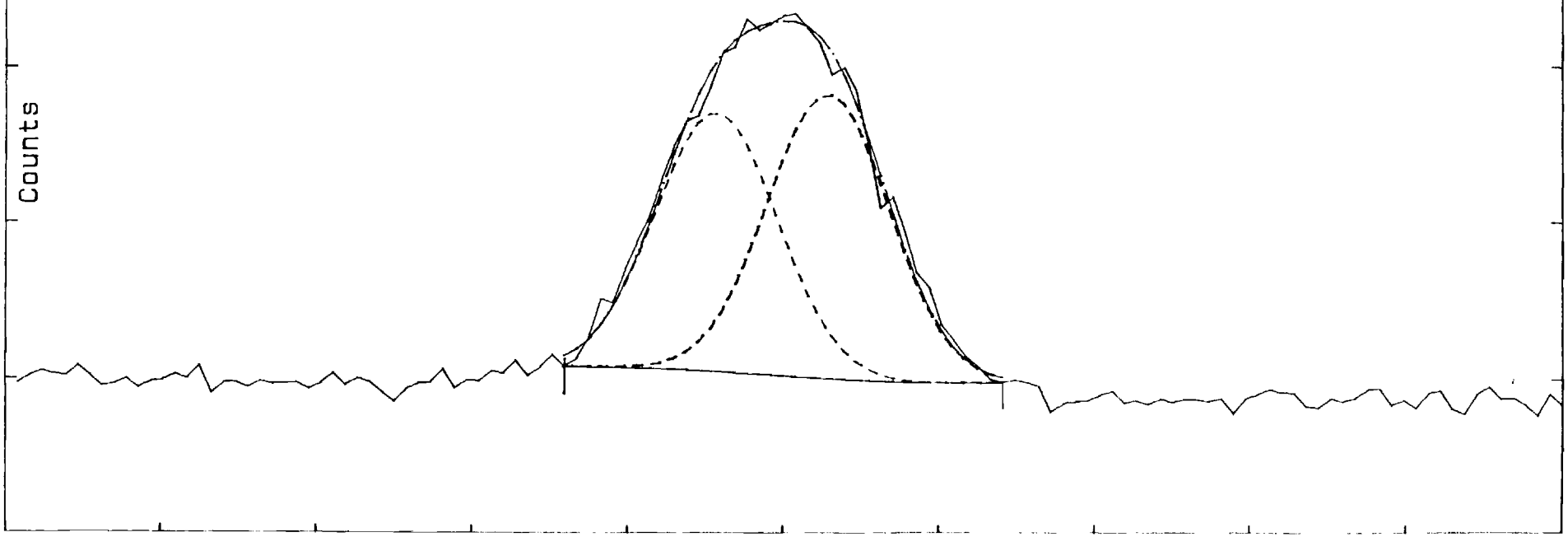
File: PPROPMRS02	Date: 8/26/1988	Spot Size: 300 u	Flood Gun: 3.0 eV
Region 3	Disc: NASA02	# of Scans: 10	Resolution: 2

Description: POLYPROPYLENE-11, O 1s SPECTRUM, PLASMA EXPOSED: 45 DEG C, 100 W, 90 MIN., NEUTRALIZATION SCREEN

Operator: WBC

2000

Energy	Width	Area	%
526.41	1.84	9131	52.4
527.86	1.84	8304	47.6



537.0 Binding Energy (eV) 517.0

Report #: MSFC-02